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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

eneral reviews of progress, reagents and methods of general application.

3. **Developments in the analytical service to industrial atomic energy.** F. J. Woodman, Clinton, W. Fletcher and G. A. Welch A.E.A., Risley, Lancs.). Second U.N. Inter- al Conference on the Peaceful Uses of Atomic y, Geneva 1958, Rep. A/CONF.15/P/1453, —Some of the advances in reducing the time alytical work which have been effected as a of development work carried out in the ical laboratories of the Industrial Group of K.A.E.A. are described. Continuous uranium ors for raffinates based on a solvent extraction tion and an absorptiometric finish and for s streams based on "in line" γ-absorption rement are discussed. For laboratory appli-, the instruments and techniques described e—a direct-reading high-resolution spectro- for U isotopic analysis; adaptations of mental techniques under high activity con-s; the use of γ-spectrometry for rapid, c determinations on large numbers of bio- l samples; the use of gas chromatography in alysis of corrosive gas mixtures.

NUCL. SCI. ABSTR.

9. **o-Dithiols in analysis. VIII. Use of the complex of toluene-3:4-dithiol in the field- g of ores and minerals.** R. E. D. Clark and Tamale-Ssali (Dept. of Sci. and Technol., Coll. and Sch. of Art, Cambridge, England). *st*, 1959, **84**, 16–23.—The zinc complex of ne-3:4-dithiol is recommended as a reagent sting for Cu, Ag, Au, Zn, Sn, Pb, V, As, Sb, o, W, S (free), Se, Te, Mn, Fe, Co and Pt in ores and minerals. Almost all the tests bed can be carried out on the coarsely crushed 30 to 60 sec., except those for the noble metals 3 min.). The tests are suited to field condi- Filtration, when necessary, is effected by g a cotton-wool plug through the liquid, the liquid above the plug being removed by e. If the solid is required, the plug is washed water by the same procedure, then dried, and st is applied directly to the enmeshed solid. reparation of Zn-free dithiol for use in tests for described. Interferences are rare and can y be avoided. A. O. JONES

0. **Dithio-oxamide as a colorimetric reagent in etection of metal ions.** Samir K. Banerji and K. Dey (Univ., Allahabad). *J. prakt. Chem.*, **6**, 62–64 (in English).—Two or three drops of % soln. of dithio-oxamide in alcohol were added nl of a 0·1 *M* soln. of the metal salt in acid, al or alkaline soln. Sensitive colour tests were ned with Cu^{2+} (olive green), Ni^{2+} (violet), (violet in alkaline soln.), and Co^{3+} (brown in acid and neutral soln., black - green in alkaline soln.). A violet - blue colour was obtained with $K_2Cr_2O_7$. CHEM. ABSTR.

4241. **Use of sulphathiazole in inorganic analysis.** Yu. D. Gnesin. *Uch. Zap. Pyatigorsk. Farmatsevt. Inst.*, 1957, **2**, 283–295; *Ref. Zhur., Khim.*, 1959, (3), Abstr. No. 7960.—The reactions of Cu^{2+}, Ag^{+}, Hg^{2+}, Hg_2^{2+}, Bi^{3+} and Co^{2+} with sulphathiazole are studied. The detectable minima (in μg) and limiting dilutions for these ions are—Cu^{2+}, 0·075 (1 in $1{\cdot}3 \times 10^7$) at pH 1·2; Ag^{+}, 0·085 (1 in $1{\cdot}1 \times 10^4$) at pH 4·6; Hg_2^{2+}, 0·07 (1 in $1{\cdot}4 \times 10^4$) at pH 4·2 to 4·6; Hg^{2+}, 0·073 (1 in $1{\cdot}3 \times 10^4$) at pH 7·8 to 8·0; Bi^{3+}, 0·43 (1 in $2{\cdot}3 \times 10^4$) at pH 4·72 to 4·98; and Co^{2+}, 0·1 (1 in 1×10^4) at pH 1·6 to 1·8 or 4·4 to 5·6. The composition and properties (m.p., behaviour on heating, and solubility in acids, alkalis and organic solvents) of the resulting compounds are studied. The structural formulae are derived from the metal contents determined.

C. D. KOPKIN

4242. **Gallein as an analytical reagent. I. Lead galleinate, its composition and analytical applications.** Ping-Shih T'ien and K'uei Wang. *Acta Chim. Sinica*, 1958, **24** (6), 407–412.—Gallein forms a blue coloration or ppt. with lead ions; the sensitivity is 0·2 μg, and Cu, Ce, Sb, Sn^{II} and Sn^{IV} interfere. The composition of the lead galleinate formed was determined by heterometric titration and by the slope-ratio method. The ratio of reagent to lead in the compound formed in an alcoholic medium was found to be 2:3, while in an aq. medium of pH 8 it was 1:1. Gallein may be used as the reagent in the heterometric titration of lead and in its colorimetric determination. Large amounts of other salts impair the determination. Beer's law is obeyed in the concn. range 5 to 150 μg of Pb per 10 ml. Gallein may also be used in the standard colorimetric series for the visual colorimetric determination of lead. SCI. ABSTR. CHINA

4243. **Simple method for recovering molybdenum from molybdenum-containing filtrates from phosphoric acid determinations.** H. Dietrich and P. von Polheim (Staatl. Landwirtschaftl. Versuchs- und Forschungsanst, Augustenberg, Post Grötzingen bei Karlsruhe, Germany). *Z. anal. Chem.*, 1959, **166** (1), 18–23.—The recovery of 85 to 90% of the Mo as pure ammonium molybdate fit for immediate use in phosphate determinations can be achieved by the proposed method. The filtrate is neutralised with aq. NH_3 and the Mo is pptd. from the boiling soln. with the calculated amount of ammonium phosphate. The soln. is set aside overnight and the ppt. is filtered off and washed with H_2O. The ppt. is dissolved in aq. NH_3 and sufficient $MgCl_2$ is added to precipitate the P. When the filtered soln. is made just acid with acetic acid, ammonium molybdate crystallises out overnight.

G. BURGER

4244. **Notes on perchloric acid and its handling in analytical work.** Analytical Methods Committee, Society for Analytical Chemistry. *Analyst*, 1959, **84**, 214–216.—In this report, prepared by the Metallic Impurities in Organic Matter Sub-committee, the circumstances in which the use of $HClO_4$ in analytical work and its storage under laboratory conditions may involve danger are described, and routine safeguards in laboratory practice to minimise or remove such danger are suggested. A. O. Jones

4245. **Analytical application of -onium compounds. XII. Tetramethylammonium phosphate buffer and its polarographic application, with some notes on the carbon dioxide effect.** Mutsuaki Shinagawa, Hiroshi Matsuo and Nobuko Okamoto (Univ., Hiroshima). *J. Sci. Hiroshima Univ., Ser. A*, 1957, **21**, 131–137.—The use of the buffer as a supporting electrolyte at negative potentials is described. Its max. buffer capacity is at pH 6·9. An obstructive wave, formed at −2·0 to −2·4 V, is believed to be due to the formation of methyl carbamate by CO_2. Boiling or treating the buffer soln. with an anion-exchange resin eliminates the wave. Chem. Abstr.

4246. **Separation of bivalent from tervalent cations in the presence of phosphate by a modified ammonia method, and comparison with separation by the urea method.** F. Krleža (Lab. of Anal. Chem., Philosoph. Fac., Univ., Sarajevo, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (4), 231–235 (in English).—The separation of Mg, Ca, Sr and Ba from Al, Cr or Fe can be achieved by pptg. the tervalent ions from a soln. containing NH_4NO_3 and enough aq. NH_3 to give a pH of 6·9 to 7·0. The maximum proportion of phosphate allowable before co-pptn. of the bivalent ion occurs has been determined, and is less than for the urea method. For a given tervalent ion, the upper permissible limit of phosphate content falls from Mg to Ba, *i.e.*, in the order of decreasing solubility product of the secondary phosphate of the bivalent ion. For a given bivalent ion, the upper permissible limit of phosphate content falls in the order Al, Fe, Cr, *i.e.*, in order of increasing solubility product of the tervalent metal phosphate. *Procedure*—Dissolve 10 g of NH_4NO_3 in 80 ml of water and boil. Add sufficient freshly distilled aq. NH_3 (1 to 9) to give a pH of 6·9 to 7·0 in the final soln. To the hot soln. add dropwise 50 ml of test soln. Cover the beaker and set aside for 20 min. at 60°, washing the ppt. three times by decantation with 100-ml portions of 0·5% aq. NH_4NO_3 soln. Allow to cool to room temp. Measure the pH of the soln. and filter off the ppt. Determine the bivalent metal in the filtrate. Figures quoted of the results obtained indicate excellent recovery of bivalent metal within the limits of phosphate content referred to above. H. M.

4247. **Indicators for non-aqueous acid - base titrimetry. I.** J. T. Stock and W. C. Purdy (Dept. of Chem., Univ. of Connecticut, Storrs, U.S.A.). *Chemist Analyst*, 1959, **48** (1), 22–28.—The literature up to June 1958 is surveyed, with 184 references. After a short discussion of the nature and history of the technique, the various procedures are briefly described, under indicator headings, giving the substance determined, titrant and solvent used. The indicators considered are azo dyes (*p*-aminoazobenzene, azo violet, methyl orange, methyl red, methyl yellow, and others less commonly used) and triphenylmethane dyes [crystal violet, r violet, malachite green and Victoria blue F Basic Blue 11)]. R. E. Ess

4248. **New applications of the diphenyl indicator in volumetric oxygen-adsorption rea** F. Sierra and G. Asensi. *An. Real Soc. Esp Quím., B*, 1958, **54** (1), 13–28.—Iodide c determined volumetrically with $AgNO_3$ i presence of Br^- or Cl^-, and Ag^+ with Br^-, or SCN^-, with a mixture of H_2SO_4, diphenyl and potassium metavanadate as indicator. Chem. Abs

4249. **Analysis under ultra-violet light. I. dala red as a fluorescence indicator in precipi titrations.** J. Bognár (Tech. Univ. for Heavy Miskolc). *Acta Chim. Acad. Sci. Hung.*, 19 (4), 433–440 (in German).—In neutral or al soln., Magdala red (C.I. Basic Red 6) is red by light and fluoresces yellow under u.v. light. colour changes to violet, and the fluoresce quenched, at a pH between 2 and 3·6. The fl cence is also quenched by the presence of Magdala red has been used successfully unde light to indicate the end-point in the titrati halogen or SCN^- with $AgNO_3$, and in the tit of Ag^+ with NaBr or KSCN. In the determi of Ag^+, 1 ml of 1% dextrin soln. is added protective colloid. The indicator is used 0·2% aq. soln. [See also *Magyar Kém.* 1959, **65** (3), 123.] G. Bur

4250. **Eriochrome black T and its calciun magnesium derivatives.** H. Diehl and F. Lind (Iowa State Coll., Ames, U.S.A.). *Anal.* 1959, **31** (3), 414–418.—Eriochrome black T Mordant Black 11) (**I**) forms only 1 to 1 pounds with Ca and Mg in the pH range 8 This and the stability constants obtained for complexes confirmed the work of Schwarze *et al.* (*Helv. Chim. Acta*, 1948, **31**, 678). measurements were made at constant ionic str with the crystalline dimethylammonium salt (preparation described). G. P. Co

4251. **Polarographic behaviour of Variamine** E. Bányai and P. Zuman (Polarographic Acad. Sci., Prague). *Coll. Czech. Chem. Com* 1959, **24** (2), 522–525 (in German).—The reve two-electron oxidation of Variamine blue Azoic Diazo Component 35) (**I**) to *p*-ani benzoquinonedi-imine has been studied p graphically in relation to pH, with the H reference electrode and K_2CrO_4, $K_3Fe(CN)_6$, and Fe^{3+} as oxidants. The oxidation of **I** K_2CrO_4 and $K_3Fe(CN)_6$ is quant. at pH 4·7 o whereas that with Ce^{4+} and Fe^{3+} is not compl acetate medium. The oxidation of **I** in borate soln. (pH 9) has been studied by the amperor titration of **I** with $K_3Fe(CN)_6$. J. Zy

4252. **Photometric determination of ind end-points in complexometric titrations.** Sô Musha, Makoto Munemori and Kin'ya C (Appl. Chem. Dept., College of Engng, Uni Osaka Prefecture, Sakai, Osaka). *Bull.* *Soc. Japan*, 1959, **32** (2), 132–137 (in Englis The utility and limitations of a graphical m (Musha *et al.*, *Ibid.*, 1957, **30**, 675) for determ the end-point in complexometric titrations photometric technique are discussed with re to the titration of Mg and Ca with EDTA (disc salt), with Eriochrome black T as indicator, a Ca with murexide as indicator. I. Jo

3. Composition of a tetra-alkylammonium base . M. L. Cluett (E. I. du Pont de Nemours , Wilmington, Del., U.S.A.). *Anal. Chem.*, **31** (4, Part I), 610–611.—The strongly basic : prepared by the reaction of tetra-*n*-butylnium iodide with Ag_2O in anhyd. methanol en found to consist of a mixture of hydroxide ıethoxide (1:1). G. P. Cook

4. Quantitative anodic chronopotentiometry at ıtinum electrode. J. D. Voorhies and J. S. ıs (Res. Div., Amer. Cyanamid Co., Bound , N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (4,), 516–519.—Anodic chronopotentiometry at ght platinum-wire electrode has been used for termination of oxidisable organic compounds. ptobenzothiazole can be determined with a e precision of ±1%, based on 13 assays in a range of 0·8 to 1·1 × 10^{-2} *M* at two current The determination of toluene-2:4-diamine ;o been studied. K. A. Proctor

5. Determination of the solubility of organic ts in mineral acids. D. G. Tuck (Cornell Ithaca, N.Y., U.S.A.). *Anal. Chim. Acta*, **20** (2), 159–163.—Hydrochloric acid is saturvith di*iso*propyl ether (**I**) by shaking and the of **I** is separated. An aliquot of the aq. is neutralised with NaOH soln. (25%, w/w), ıuid is centrifuged in a Babcock tube and the f **I** that separates is read on the previously ıted neck of the tube. A correction for the lity of **I** in the salt soln. is determined. The lity of **I** in aq. HCl rises sharply when the of the acid exceeds 7 *M*. H. N. S.

6. Applications of extraction in inorganic sis. A. K. Babko and F. G. Zharovskiĭ. . *Lab.*, 1959, **25** (1), 42–52.—A review is ıted, with 166 references. G. S. Smith

7. Infra-red analysis of solid substances. ew. G. Duyckaerts (Univ. of Liége, Belgium). *st*, 1959, **84**, 201–214.—The physical and cal factors affecting the i.r. spectra of solid ıances are reviewed. Among the physical s discussed are the influence of the dimensions individual absorbing particles and of differin refractive index between the solid absorbing ıance and the dispersing medium (liquid in, KBr, etc.). The chemical influences sed include the adsorption of the substance on les of the dispersing medium, the chemical on or complex-formation of the substance the medium, the polymorphism of the sube, mixed crystal formation and distortion of ystalline structure. (23 references.) A. O. Jones

8. The method of oscillographic polarography mical analysis. J. Doležal and J. Zýka (Inst. Chem., Charles' Univ., Prague). *Ost. Chem.* 1959, **60** (7), 189–196.—Applications of ɔgraphic polarography are reviewed and the ıatus used is described.

9. Radioactivity applied to analytical chem- H. Götte (Max Planck Inst. f. Chem., Mainz, any). *Técnica, Lisbon*, 1959, **34**, 525–535.— lew with 9 references.

0. Radiochemical investigations on the re- for analysis of trace elements in organic and ical materials. Report to the Analytical ods Committee. T. T. Gorsuch (Radiochem. Centre, White Lion Rd., Amersham, Bucks., England). *Analyst*, 1959, **84**, 135–173.—The radiochemical methods used involve the final determination of radioactive isotopes either added as tracers or formed by irradiation of the sample. Since any of the elements extraneously introduced during the analysis will be inactive, measurement of the radioactivity of the separated element will be a true indication of the loss of the element caused by the conditions of the analysis. Also, adsorption of the element by co-pptn. or by the apparatus can be located and measured. For stated reasons, cocoa was generally used as the sample and the investigation was then extended to some other materials. The recovery of the following elements under various conditions of analysis was investigated —Pb, Hg, Zn, Se, As, Cu, Co, Ag, Sb, Cr, Mo, Sr and Fe. A. O. Jones

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

4261. **Reductimetric determination of metals in non-aqueous media.** J. Minczewski, S. Kołyga and L. Wódkiewicz (Polish Acad. Sci., Warsaw). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/1596, 8 pp.—Chromous acetate in dioxan soln. was used as a reducing agent in non-aq. media. The method was applied to the determination of uranyl nitrate in a soln. of ethanol and to the determination of Cu and V after extraction with a soln. of 8-hydroxyquinoline in $CHCl_3$. Good reproducibility and accuracy were obtained in concn. of ≃0·2 mg per ml.

Nucl. Sci. Abstr.

4262. **Indirect ascorbimetric determination of strong oxidising agents.** L. Erdey, I. Buzás and K. Vigh (Inst. f. Allg. Chem., Tech. Univ., Budapest). *Period. Polytech.*, 1959, **3** (1), 1–15 (in German).—Chlorite, ClO^-, BrO^-, $S_2O_3^{2-}$, $Cr_2O_7^{2-}$, MnO_4^-, Cl, Br and H_2O_2 can all be determined by adding an excess of Fe^{II} salt and titrating the Fe^{III} formed with standard ascorbic acid. Nitrites may be titrated potentiometrically with ascorbic acid in an acetate buffer at pH 4.

T. R. Andrew

4263. **Separation of zinc, lead and copper on an anion-exchange resin.** M. N. Zvereva and V. N. Shibarov. *Vestn. Leningr. Univ.*, 1959, No. 4, *Ser. Fiz. i Khim.*, (1), 145–148.—Copper, Zn and Pb were separated quant. by means of anion-exchange resins PE-9 and EDE-10 "P." The air-dried anionite (6 g) was supported in a column 1·0 to 1·5 cm in diam. and the exchange carried out in $CHCl_3$. Separation was commenced with 2 *N* HCl (100 ml) which rapidly removed the Cu but left the Pb and Zn complexes on the column. Zinc was then removed with 0·3 *N* HCl, and Pb with H_2O. The tabulated results indicate that the accuracy of determination of Cu, Zn and Pb is not greater than that of the usual analytical procedures.

K. R. C.

4264. **Use of tiron for the chromatographic separation of iron, aluminium, titanium, tin and antimony from calcium, magnesium, zinc and**

copper. I. R. N. Golovatyĭ. *Ukr. Khim. Zhur.*, 1958, **24** (5), 653–655; *Ref. Zhur., Khim.*, 1959, (11), Abstr. No. 38,274.—The method described is based on the formation of complexes of Fe^{3+}, Al^{3+}, Sb^{3+}, Sn^{4+} and Ti^{4+} with tiron (**I**), which are not decomposed when the soln. is passed through a column of cationite SBS in the NH_4^+ form. The complexes of Ca^{2+}, Mg^{2+}, Mn^{2+}, Zn^{2+} and Cu^{2+} are less stable, and these cations are retained on the resin. *Procedure*—Treat the test soln. with sufficient 0·05 *M* **I** to complex Fe^{3+}, Al^{3+}, Sb^{3+}, Sn^{4+} and Ti^{4+} completely. If the soln. contains Sb^{3+} or Sn^{4+}, the formation of the complexes is accelerated by heating to boiling-point. Then add aq. NH_3 to the soln. till the blue colour of the complex of Fe^{3+} with **I** changes to red (pH >8); if the soln. does not contain Fe, a small amount is added. After adjusting the pH, dilute the soln. to between 120 and 150 ml and pass it through the column at a rate of 3 to 4 ml per min. Calcium, Mg, Cu, Zn and large amounts of Mn are eluted from the column with hot 3 *N* HCl, and small amounts of Mn with cold 1·5 *N* HNO_3. Magnesium, Zn and large amounts of Mn are determined in the filtrate with phosphate, Ca with oxalate, and Cu iodimetrically; small amounts of Mn and Cu are determined photometrically. C. D. Kopkin

4265. **Extraction in the radiometric titration of certain cations.** I. M. Korenman, F. R. Sheyanova, L. V. Gorbunova and N. K. Romanova. *Trudy Khim. i Khim. Tekhnol.*, 1958, (1), 109–117; *Ref. Zhur., Khim.*, 1959, (2), Abstr. No. 4289.—The use of ^{65}Zn and ^{115}Cd as non-isotopic indicators in the radiometric titration of Hg, Cu, Zn and Cd with a $CHCl_3$ soln. of dithizone (**I**) is described. Conditions are studied for the radiometric analysis of the binary mixtures Hg - Zn, Hg - Cd, Cu - Zn, and Cu - Cd, and the ternary mixtures Hg - Cu - Zn and Ag - Cu - Cd. The use of ^{65}Zn and ^{115}Cd as non-isotopic indicators for the determination of Hg and Cu enables preparations of low specific activity to be used, since they may be added to the soln. in amounts commensurate with those of the ions to be determined, and avoids the use of short-lived isotopes of Hg and Cu. For the simultaneous determination of Hg and Zn (or Cd) as the dithizonates by the radiometric method (*Ref. Zhur., Khim.*, 1958, Abstr. No. 24,729), add to a series of tubes (with ground glass stoppers) 0·5 ml of the soln. of Hg, 0·5 ml of a soln. of a zinc salt of known concn. labelled with ^{65}Zn (of specific activity 35 impulses per min. per μg), and add 1 ml of a buffer soln. of pH 4·7, various amounts of a $CHCl_3$ soln. of **I**, and pure $CHCl_3$ to give a total $CHCl_3$ layer of 2 ml. Shake the contents of the tubes for 15 min., centrifuge for 1 or 2 min., and determine the activities of the aq. and $CHCl_3$ layers in each tube. By the gradual addition of the soln. of **I**, mercury dithizonate is first formed and extracted (instability const. 7×10^{-43}); the activity of the aq. and $CHCl_3$ layers remains constant. As soon as zinc dithizonate (instability const. 8×10^{-24}) or cadmium dithizonate (instability const. $2{\cdot}9 \times 10^{-26}$) begins to be extracted, the activity of the aq. layer decreases, while that of the $CHCl_3$ layer increases. The total activity of equal vol. of the aq. and $CHCl_3$ layers should be constant at all points of the titration. The end-point is determined graphically or from formulae derived from the solution of the simultaneous equations of the titration curves. The concn. of the soln. of **I** is found in the same way, using known amounts of Hg and Zn. The described procedure may be used for the determ tion of micro amounts of Hg. An analo method, based on the differing stabilities of dithizonates, permits the determination of Cu Zn (or Cd) without a preliminary separation, also enables a mixture of three cations to analysed (*e.g.*, Hg - Cu - Zn and Ag - Cu - Cd) this case use as indicators isotopes of the elem which form the least stable and the most st complexes (*e.g.*, Hg and Zn). Satisfactory re are obtained when applying the developed cedure for the radiometric titration of two ca to the analysis of brass, duralumin and a alloy (with ^{65}Zn as indicator). C. D. Kopk

4266. **Simple method for determination of radiochemical purity of isotopes by means of p electrophoresis.** M. Mach (Res. Inst. of Radio Prague). Second U.N. International Conferenc the Peaceful Uses of Atomic Energy, Geneva, Rep. A/CONF.15/P/2109, 10 pp.—It was f that paper electrophoresis ensures a better sep tion of active contaminants than paper chrom graphy. With the acid electrolytes $HClO_4$ HNO_3, an absolute separation of orthophosp sulphate and chloride was achieved, *i.e.*, of forms, in which the impurities of radio-isot ^{35}S, ^{36}Cl, and of irradiated chlorides as a resu production processes are present. Trace amo of sulphates were separated from phosphates dil. HCl as electrolyte. The separation of r tellurium from radio-iodine was achieved after the oxidation of samples by using diff electrolytes at the positive and negative electr By using the results of Zimakov *et al.*, contamin ruthenium was separated from Sr and Y in 2% of $K_4Fe(CN)_6$ and contaminating radio-cae from ^{90}Sr in 0·5 *N* NaOH by paper electropho (*Cf. Anal. Abstr.*, 1959, **6**, 2403.)

Nucl. Sci. Abst

4267. **Radiometric determination of thal silver and lead by the non-isotopic indicator me** I. M. Korenman, F. R. Sheyanova and F Mulyanov. *Trudy Khim. i Khim. Tekhnol.*, (1), 118–124; *Ref. Zhur., Khim.*, 1959, (2), A No. 4290.—Conditions are studied for the r metric titration of Tl with a soln. of KI, ^{204}Tl as radioactive indicator; procedures are described for the determination of Tl, Ag and with ^{204}Tl and ^{110}Ag as indicators. To deter Tl, add to each of several centrifuge tubes 1 a soln. of a thallium salt (4·09 mg of Tl) conta ^{204}Tl (0·3 μg of Tl) of specific activity 1 counts per min. per μg, acidify the soln. with drops of 2 *N* HNO_3, add a small amount of wat that after the titration the final vol. will be 2· add various amounts of 0·1 *N* KI, rub the tube with a glass rod, centrifuge off the ppt. of extract 0·2 ml of the centrifugate and measu activity. The end-point is found graphically the activity at two points during the titra Completely satisfactory results are obtaine determining 0·4 to 4 mg of Tl per ml; with sm amounts of Tl unsatisfactory results are obta because of the solubility of TlI. It is shown th analogous titration procedure may be used fo determination of Ag and Tl when present toge the activity of the test soln. remains constant d the titration of Ag and decreases with the titr of Tl. The titration of a mixture of Ag and possible with ^{110}Ag as indicator: Tl is ppt described above with KI soln. in tubes of a s shape, a known vol. of a soln. of $AgNO_3$ labelled

s added, the soln. are mixed, and the activity action of the mother liquor is measured (with ometric titration of the residue). To deter- Pb, to 1 ml of a soln. of $Pb(NO_3)_2$ add 0·2 ml n. of the thallium salt (0·3 μg of Tl) containing acidify with two drops of acetic acid and with a soln. of K_2CrO_4, measuring the y of a fraction of the centrifugate. The ed method for determining Pb gives satis- results and is more convenient than the d in which the short-lived ^{212}Pb is used as tor. C. D. KOPKIN

. Induced radiation analysis for silicon, alu- n and sodium in igneous rocks. G. M. ell, K. Bramadat, R. A. Knutson and A. C. ck. *Trans. Roy. Soc. Canada, Sect. IV*, **51**, 19–31.—Rapid quant. determination of and Na in rocks can be made by comparing the ity of these rocks with that of chemically ed rocks. The induced radiation is propor- to the mass of the elements and is measured cintillation counter. Coarsely crushed rock)-g samples) was subjected to fast neutrons to ine the Si content, thermal neutrons for the ntent, and slow neutrons for longer periods termine the Na content. The degree of cy was found to be within ±1·75% for SiO_2, % for Al_2O_3, and ±0·3% for Na_2O. CHEM. ABSTR.

). Micro-determination of water by titration Fischer reagent. E. L. Bastin, H. Siegel and Bullock (Shell Devel. Co., Emeryville, Calif.,). *Anal. Chem.*, 1959, **31** (3), 467–473.— s of a special apparatus and of its use are for the determination of quantities of H_2O range 0·01 to 3 mg. H. F. W. KIRKPATRICK

). Non-dispersive infra-red analysis of mixtures er and heavy water. C. A. Bosselaar (Stichting Fundamenteel Onderzoek der Materie, nt). Second U.N. International Conference e Peaceful Uses of Atomic Energy, Geneva, Rep. A/CONF.15/P/2250, 7 pp.—The deu- n content can be determined by means of a ispersive i.r. gas analyser. An apparatus is bed to measure the D_2O content of vapour res from the natural concn. range to 40% of with an accuracy of about 0·5% of D_2O. better results can be obtained with liquid res. The method is suitable for rapid deter- ions on flowing streams of water. NUCL. SCI. ABSTR.

1. Determination of lithium in lithium - sium alloys. D. H. Wilkins (General Electric New York, U.S.A.). *Anal. Chim. Acta*, 1959, 2), 116–118.—*Procedure*—Dissolve a 0·5-g e in dil. HCl, evaporate to dryness and dis- the residue in water with sufficient dil. HCl oduce a clear soln. Dilute to 100 ml and er 10 ml to a column (1 cm × 50 cm) of ly acidic cation-exchange resin (H^+ form, 100) mesh), washing the soln. into the resin with . Elute the Li^+ with 150 ml of 0·5 *N* HCl. orate the eluate to dryness, dry the residue at dissolve it in water and titrate with standard O_3 soln. To regenerate the column, elute the with 4 *N* HCl. H. N. S.

2. Complexometric determination of sodium. lasek and M. Dugandžić (Univ., Graz, Austria). *chim. Acta*, 1959, (3), 488–489.—In the titra- of Zn contained in the ppt. of sodium zinc uranyl acetate, dithizone is used as indicator in place of Eriochrome black T. Even when using 0·001 *M* EDTA, a sharper change is observed. Very good recovery is shown within the range from 80 to 318 μg of added Na. D. F. PHILLIPS

4273. **Determination of sodium from the zinc content of the triple uranyl acetate compound by extraction titration.** L. V. Markova and K. E. Kleĭner (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (2), 144–145.—The sodium zinc uranyl acetate compound is pptd., washed and dissolved, and the Zn content of the soln. is determined by dithizone extraction titration (Sandell, *Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 464). The determination of Na from the Zn content is satisfactory in the presence of 50 times as much K as Na. G. S. SMITH

4274. **Separation and determination of traces of sodium present in potassium salts.** J. Angot (Soc. d'Études Chim. pour l'Ind. et l'Agric., Argenteuil, Seine-et-Oise, France). *Mikrochim. Acta*, 1959, (3), 346–356 (in French).—A preliminary separation of the trace amount of Na from K is carried out chromatographically on an ion-exchange column of Amberlite IR-120 (H^+ form). The Na is eluted with 0·5 *N* HCl and the determination completed by flame photometry by using a very simple flame photometer in which the characteristic line is isolated by means of an interference filter. The excitation flame is fed by air - propane or air - butane. The absence of interfering ions facilitates the establishment of ideal conditions for the flame photometry. The determination of 0·05% of Na in potassium salts with a relative precision of $\simeq \pm 5\%$ has been achieved, but it is envisaged that, by the use of certain special precautions (*e.g.*, the use of quartz for the ion-exchange tube and its accessories, a laboratory with a dust-free atmosphere and rigorous purification of reagents), it should be possible to determine 0·001% of Na with a relative precision of ±10%. D. F. PHILLIPS

4275. **Determination of potassium and sodium in mixtures of their salts by means of cation exchangers.** G. Gabrielson (Elektrokemiska AB, Bohus, Sweden). *Anal. Chim. Acta*, 1959, **20** (2), 146–151.—To determine NaCl and KCl in a soln. of the two salts a portion is first evaporated to dryness to find the total content. Then a similar portion is passed through a column of Amberlite IR-120, charged with K^+, to exchange Na^+ for K^+, the resulting soln. is evaporated to dryness and the NaCl is calculated from the gain in wt. over the original content. Alternatively, the KCl may be determined by exchanging K^+ for Na^+ and calculating from the loss in wt. In another less accurate procedure, both Na^+ and K^+ are exchanged for H^+ and the resulting soln. is titrated with standard NaOH soln. This titre, and the total wt. of dry salts determined in a similar sample, provide the data for the calculation of the components. H. N. S.

4276. **Spectrophotometric micro-determination of potassium and sodium.** F. Kreisky (Pharmakol. Inst., Univ., Gotenburg, Sweden). *Mikrochim. Acta*, 1959, (2), 243–248 (in German).—Potassium in tissues in the concn. range of 4 to 40 μg can be determined as potassium iodoplatinate and Na (3·2 to 32 μg) as sodium zinc uranyl acetate. *Procedure for potassium*—The sample (8 to 80 μg of KCl) in 0·01 *N* HCl is treated with 0·1 ml of 0·5% chloroplatinic acid in a calibrated 15-ml centrifuge

tube, placed in a water bath and evaporated to dryness at 90° to 95°. When cool, the residue is first moistened with 1 ml of abs. ethanol, then a further 10 ml is added and the mixture is centrifuged for 20 min. After decantation it is washed with ethanol (1 × 7 ml, 3 × 5 ml), being centrifuged each time for 12 to 15 min. Last traces of ethanol are removed at 90° to 95°, 6 ml of *N* HCl is added, and the soln. is transferred to a 10-ml flask and set aside overnight. To an aliquot containing 0·6 to 6 μg of KCl per ml is added 0·5 ml of KI soln. (5%), the mixture is made up to 5 ml with *N* HCl and set aside for 4 hr. in the dark. The extinction is measured at 496 mμ. A blank determination is arried out at the same time. *Procedure for sodium* —The sample (0·5 to 1 ml in 0·01 *N* HCl, containing 6 to 80 μg of NaCl per ml) is placed in a 15-ml calibrated centrifuge tube and evaporated in a water bath. When the residue is cool, 0·08 ml of trichloroacetic acid (2%) is added followed, in subdued light, by 1 ml of zinc uranyl acetate soln. (specified) and 0·5 ml of abs. ethanol. After being set aside overnight, the mixture is centrifuged for 30 min., then decanted, washed with acetone (2 × 5 ml) and centrifuged each time for 20 min. The acetone is evaporated at 90° to 95° for 12 min. and 6 ml of Na citrate soln. (0·4%) is added. The soln. is transferred to a 10-ml flask, washed with 4 ml of Na citrate soln. and made up to 10 ml with Na citrate soln. After 2·5 hr. the extinction is measured at 224 and 244 mμ. The accuracy is within ± 5·5% for K for 4 to 8 μg and ± 2% for 20 to 80 μg, and ± 1·5% for Na. B. B. Bauminger

4277. **Determination of alkali hydrogen carbonates in the presence of alkali carbonates.** E. Bakács-Polgár and L. Szekeres (Inst. f. Chem. d. Tierärztl. Hochsch., Budapest). *Pharm. Zentralh.*, 1959, **98** (1), 3–5.—Add a known excess of NaOH soln. to the mixture (containing approx. equal amounts of HCO_3^- and CO_3^{2-}), shake vigorously with a slight excess of $BaCl_2$ soln., set aside for a few minutes and titrate with $ZnCl_2$ soln., with phenolphthalein as indicator (*cf. Anal. Abstr.*, 1958, **5**, 3265).
A. R. Rogers

4278. **Analysis of soda ash (anhydrous sodium carbonate). Proposed revision of TAPPI standard T612m-44.** *TAPPI*, 1958, **41**, 167A-168A.—In this revision, apparatus, reagent and report sections have been added and a weighing bottle substituted for a weighing tube for measuring the loss of weight at 150°. *Total alkalinity*—A 250-ml portion of the soln. (corresponding to 2·5 g of the specimen) is titrated quickly with 0·5 *N* HCl, with 3 drops of methyl orange as indicator. To another 250 ml about 1 ml less of 0·5 *N* HCl is added, CO_2 is removed by boiling and the titration completed after cooling. *Hydroxide*—To 250 ml of soln. is added 100 ml of 10% $BaCl_2$ soln. and the ppt. is filtered off. The filtrate is titrated with 0·5 *N* HCl to phenolphthalein. *Bicarbonate*—A 10-g portion of solid in 50 ml of H_2O is titrated with 0·5 *N* NaOH until one drop produces an immediate dark coloration with $AgNO_3$ soln. Bicarbonate cannot be present together with hydroxide. *Carbonate*—This component is calculated from the bicarbonate and total alkalinity values.
R. Aicher

4279. **Determination of free potassium metal in coloured potassium chloride.** L. Silverman and M. E. Shideler (Atomics International Div., N. Amer. Aviation, Inc., Canoga Park, Calif.). *U.S. Atomic Energy Comm.*, Rep. NAA-SR-1421, 1956, 13 pp.—The free K reacts with water to form K and the hydroxyl ion concn. is determined b change. A particularly useful pH range of 9 was selected. Coloured crystals were pro by the action of K vapour on halide crystals the experimental results of the analysis o coloured KCl crystals are shown to be withi 95% confidence level. Nucl. Sci. Abs

4280. **Determination of ammonium and pota ions in mixtures of alkali metals. Use of mercur EDTA.** F. S. Sadek and C. N. Reilley (De Chem., Univ. of N. Carolina, Chapel Hill). *Chem.*, 1959, **31** (4, Part I), 494–498.—*Proced (for NH_4^+ in the presence of Na and free N* Pipette an excess of Hg^{II} - EDTA soln. [a stoi metric mixture of acidic $Hg(NO_3)_2$ and E 0·1 *M* soln., neutralised to pH 6·8] into a flask 6 drops of bromothymol blue (0·1% in meth and 3 drops of red dye (*e.g.*, 0·1% Ponceau P H_2O) and adjust to pH 6·8 (grey colour). Ad sample soln., which gives a yellow colour, titrate with 0·1 *N* NaOH back to grey. So K and free NH_3 in 100-fold excess do not inte SO_4^{2-} and small amounts of Cl^- or F^- ca tolerated, but PO_4^{3-} must be absent. Whe Mg, Cu and Fe are present, their EDTA titre be found and allowed for. *Procedure B (for and K^+)*—Neutralise the sample (containin 10 mg of NH_4^+ and K) with pure NaOH or HC dilute to 50 ml. Add conc. HCl (0·5 ml) and of Na tetraphenylboron (**I**) soln. (3 g of **I** in 1 of H_2O, shaken with pure Al_2O_3 and filtered). and filter after 5 min. through a No. 4 or 5 sin funnel. Wash the ppt. with H_2O and dissol in 4 to 10 ml of dimethylformamide. Tra the soln. to the original beaker, add an exce Hg^{II} - EDTA soln., adjust to pH 4·5 to 6·0, sti dilute to 200 ml. Neutralise with NaOH, excess of aq. NH_3 buffer soln. (pH 10) and a drops of 0·1% Eriochrome black T soln. Ti from blue to red with standard Mg soln. procedure can be applied to other insol. compo of **I**. P. D. Parr-Richa

4281. **Rapid method for the determinatio rubidium in silicate minerals.** E. S. Burkse T. G. Kornienko. *Ukr. Khim. Zhur.*, 1958, 2 375–378; *Ref. Zhur., Khim.*, 1959, (1), Abstr 914.—The use of the radioactive isotope ^{86} proposed for establishing the losses of Rb, am ing to 20 to 50%, observed in the determinati Rb in silicate rocks by a gravimetric method. use of ^{86}Rb simplifies the method and conside shortens the time for the analysis (from 20 days to 10 days). To the sample of silicate add a known amount of ^{86}Rb and decompos sample with HF. Treat the residue with Ca(and precipitate Ca from the resulting sol alkali-metal hydroxides as carbonate and ox Remove ammonium salts and oxalic acid fro soln. of alkali-metal carbonates, and conver carbonates into chlorides. To concentrate evaporate the soln. to dryness and extract residue three times with a mixture of water (and ethanol saturated with HCl (4 ml). T separated mixture of salts add water (0·3 ml a mixture of ethanol and conc. HCl (2:1) (2· and set aside for 10 to 12 hr. to separate Then precipitate Rb from the hot ethanolic with a 40% ethanolic soln. of $SnCl_4$ and weig ppt. of Rb_2SnCl_6. Measure the β-activity o ppt. and compare it with the activity of a sta containing the same amount of ^{86}Rb as was a

rock sample, and so calculate the content of the rock. The described method has been on the determination of Rb in biotites.
C. D. KOPKIN

2. **Increase of sensitivity and accuracy of the photometric determination of caesium in als.** E. A. Fabrikova (Inst. of Mineral., em. and Crystallochem. of Rare Elements, Sci., USSR, Moscow). *Zhur. Anal. Khim.*, **14** (1), 41–44.—The presence of other alkali s, when in greater concn. than Cs, increases tensity of the line due to Cs at 8521 A; K has eatest effect; as the concn. of K increases, the ity reaches a max. which is up to 25 times than normal. The standard curve of ity *vs.* concn. of Cs (0·2 to 100 μg per ml) in resence of 2500 μg of K per ml is almost near. At the saturation intensities, the on of Na, Li or Rb has an imperceptible nce on the emission of Cs. To determine Cs, en the sample (0·5 to 1 g) in a platinum dish, onc. H_2SO_4 (0·4 ml), HF (20 ml) and HNO_3 3 drops), heat to 70° or 80° to decompose the e, increase the heating to fumes of SO_3, add water (2 ml) and conc. H_2SO_4 (0·1 ml), heat to fumes, evaporate again after adding (5 ml), dilute with 25 or 30 ml of water and to dissolve. Dilute to 70 ml, heat just to g, add powdered basic lead carbonate with ıg till alkaline to methyl red, boil for several ;es, stir and filter. Wash the residue with hot to give a total filtrate of 120 to 130 ml, ɔrate to 30 ml, and cool; filter, and make the ;e up to 50 ml. Determine K with the lines and 7699 A. To 20 ml of soln. add sufficient $_4$ soln. (0·0557 g per ml) to give, after dilution, g of K per ml, dilute to 25 ml, and determine 8521 A. The mean relative error is $\pm$4·45% ·01% and $\pm$9·7% for 0·001% of Cs. The minable minimum is 0·2 μg of Cs per ml.
C. D. KOPKIN

3. **Analytical use of pyridino-copper fumarate.** Cherkesov, A. S. Mel'nikova and T. S. Zhigal- *Trudy Astrakhansk. Tekhn. Inst. Rybn. i Khoz.*, 1958, (5), 143–146; *Ref. Zhur.*, ., 1959, (3), Abstr. No. 7969.—The interaction $^{2+}$ with a soln. of fumaric acid and pyridine of fumaric acid in 100 ml of aq. *M* pyridine) pyridino-copper fumarate, of the composition $^{\prime}y)_2]C_4H_2O_4.H_2O$ (**I**), which is insol. in water, ethanol, $CHCl_3$ and other organic solvents. solves in conc. aq. NH_3 to form the cupr- ɔnium complex, and in HCl, H_2SO_4 and HNO_3 rm the corresponding salts. On being heated 0°, **I** loses its water of crystallisation, and at r temperatures decomposes with the formation O. **I** may be used for the gravimetric deter- tion of Cu (weighed as CuO) and for the micro- alloscopic detection of Cu in the presence of lkali and alkaline-earth metals, Ni (<10 mg l), Pd (<1 mg per ml), Cd, Co, Zn, Mn and Cr. he detection, place 1 drop of test soln. on a ly warm slide and add 1 drop of reagent soln.; e presence of Cu, large blue crystals of **I** are sited from the cooled soln.; these coalesce into ns. The detectable minimum of Cu is 0·2 μg; miting dilution is 1 in 5 $\times$ 10^4. The presence , Fe and Al greatly lowers the sensitivity.
C. D. KOPKIN

4. **Rapid determination of copper and zinc ass solutions.** R. C. Armét. *Electroplating*, **12** (2), 56–57.—A rapid and accurate method consists in determining Cu by the iodine - thio- sulphate procedure, followed by determination of Zn by EDTA, with Xylenol orange as indicator. Results of analyses of different brass soln. by this process and by the electrolytic method are tabu- lated and show satisfactory agreement.
C. H. COWPER-COLES

4285. **Controlled-potential coulometric deter- mination of copper and uranium.** W. D. Shults and P. F. Thomason (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 492–494.—Solutions of mixed copper and uranium sulphates can be analysed with coeff. of variation of $\simeq \pm$ 0·1%, regardless of the Cu to U ratio. Two titrations are performed, the first, a reduction at $-$0·3 V in which both elements are reduced, and the second, a re-oxidation of the Cu alone at +0·175 V.
K. A. PROCTOR

4286. **Spectrographic determination of copper, lithium and rubidium in mineral salts.** B. O. Filonov and M. M. Pavlyuchenko. *Sb. Nauch. Rabot. Inst. Khim., Akad. Nauk BSSR*, 1958, (6), 92–101; *Ref. Zhur., Khim.*, 1959, (3), Abstr. No. 8014.—The analysis is made by the method of standard additions, the unknown concn. x being calculated from the formula $x = a^2(c - 2a)$, where a and c are the amounts added [Pavlyuchenko *et al.*, *Sb. Nauch. Rabot. Inst. Khim., Akad. Nauk BSSR*, 1958, (6), 102; *Ref. Zhur., Khim.*, 1959, Abstr. No. 986]. The sample is introduced into an a.c. arc discharge on a moving electrode. To determine Cu, a hole is made in a horizontal carbon electrode and is filled with the powdered sample. A sensitivity of 10^{-4} % is achieved by using the line Cu 3247·5 A with Ag 3286·7 A as comparison line. The samples are vaporised at 8 amp. For Li and Rb the analytical lines are Li 6707·8 A and Ba 6675·2 A, and Rb 7800·2 A and Ba 7911·35 A. The sensitivity is 5 $\times$ 10^{-5} % for Li and 5 $\times$ 10^{-4} % for Rb. The mean error of two determinations is $\pm$ 10 %. It is established that by diluting the samples of Cu and Li with NaCl and carbon powder, and by evapor- ating the substance completely from the electrode groove, the influence of third components is practi- cally eliminated and a single graph may be used.
C. D. KOPKIN

4287. **High-frequency titrations. I. The silver - thiocyanate reaction.** Bharat R. Sant and Anil K. Mukherji (Louisiana State Univ., Baton Rouge, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (2), 124–127.—The high-frequency method is applied to the titration of Ag^+ (3·2 to 6·7 mg), in concn. down to 0·0003 *M*, with KSCN. At the lowest concn. the error is $\simeq$5%. Similar accuracy is obtained in the titration of KSCN or NH_4SCN with standard $AgNO_3$ soln.
H. N. S.

4288. **High-frequency titrations of silver and thallium(I) with sodium tetraphenylboron.** Anil K. Mukherji and Bharat R. Sant (Louisiana State Univ., Baton Rouge, U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 608–610.—The pptn. of Ag or Tl^I with Na tetraphenylboron (**I**) was studied by high-frequency titration, and the stoicheio- metric formation of Ag tetraphenylboron or Tl tetraphenylboron was confirmed. The method is suitable for milligram amounts of Ag or Tl and for dilute soln. of tetraphenylboron ions, with an error of 1%. The concn. of the titrant was adjusted so that the total vol. change was $\not>$ 3%, and the end- point was found graphically. When Ag^+ or Tl^+ are titrated with **I**, the capacitance at first decreases

and then increases after the end-point. In reverse titrations, there is continuous increase in the readings, the slope of the curve being steeper after the end-point. P. D. PARR-RICHARD

4289. **Determination of gold in alloys with copper and silver by means of potentiometric titration with quinol.** A. Czapliński and K. Trokowicz (Dept. of Tech. Analysis, Polytech. Inst., Gdansk). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 463–469.—When the alloy is dissolved in aqua regia, Au is converted into $AuCl_3$ which, in the presence of an excess of HCl, changes into $HAuCl_4$, producing with NaCl a stable salt, $NaAuCl_4$, which in turn is reduced by quinol to Au according to the equation—

$$2NaAuCl_4 + 3C_6H_4(OH)_2 = 3C_6H_4O_2 + 2NaCl + 6HCl + 2Au.$$

Under strictly controlled conditions this reaction can be applied to quant. determination. *Procedure*—The sample (140 mg of Au) is dissolved in aqua regia in the presence of NaCl, evaporated nearly to dryness, cooled, treated with 10 ml of conc. HCl and the volume reduced to 1 ml by evaporation. This soln. is diluted to 100 ml with H_2O, the AgCl ppt. is filtered off and the filtrate is made up to 250 ml with H_2O. An aliquot (25 ml) is again diluted to 100 ml and titrated with 0·01 *N* quinol at room temp., with a platinum and calomel electrode pair. The potential jump at the end-point exceeds 100 mV. It is suggested that this method can be applied to gold alloys containing Cu, Ag, Sn and Cd. W. B. MIASKOWSKI

4290. **New analytical methods for beryllium.** Kenji Motojima (Atomic Energy Res. Inst., Japan). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/1325, 23 pp.—8-Hydroxyquinaldine was found to be useful as an analytical reagent for the determination of beryllium. With this reagent, Be can be determined gravimetrically, volumetrically, spectrophotometrically and even fluorimetrically. One of the main advantages of these methods lies in the elimination of Al interference, which is common in the usual Be determinations. Consequently, no preliminary separation of Al is required and a simple and accurate analytical method for Be determination is thus provided.

NUCL. SCI. ABSTR.

4291. **Fluorimetric determination of sub-microgram quantities of beryllium.** C. W. Sill and C. P. Willis (Health and Safety Div., U.S. Atomic Energy Comm., Idaho Falls). *Anal. Chem.*, 1959, **31** (4, Part I), 598–608.—By the fluorimetric method described, $\lesssim 0{\cdot}0004$ μg of Be can be detected with good precision. The permanent glass standards used (made from uranium glass spheres fused into borosilicate glass) show no decrease in fluorescence in low-intensity u.v. light. *Procedure*—To a sample of Be in 0·5 ml of $HClO_4$ add 8·2 *N* NaOH (3 ml) and 3 drops of 0·01% quinine sulphate in water containing 2% (v/v) of 72% $HClO_4$. Add $HClO_4$ sufficient to give a brilliant blue fluorescence under u.v. light; add 1 drop in excess, and transfer the soln. to a 25-ml flask. Add 0·5 ml of triethanolamine (**I**) - EDTA (5 g of EDTA and 3 ml of **I** in 95 ml of H_2O), then sufficient *N* NaOH to extinguish the fluorescence and 2 drops in excess. Add 0·5 ml of sodium stannite (1·5 g of $SnCl_2.2H_2O$ in 25 ml of H_2O, poured into 2·4 g of NaOH in 10 ml of H_2O and diluted to 50 ml) and 5 ml of buffer soln. (50 ml of piperidine added to 10 g of hydrazine sulphate and 5 g of EDTA in H_2O, and diluted to 500 ml). Mix and dilute to vol. Add 1 ml of morin (25 ml of 8 *M* $NaClO_4$ and 2 drops of 10% E soln. mixed with 7·8 mg of morin in 25 n ethanol and diluted with H_2O to 100 ml) unde surface of the soln. Mix and place in a bath trolled to within ±2° of room temp., then me the fluorescence, which is a linear function o concn. of Be up to 1 μg per 26 ml. The effec morin concn., alkalinity, the oxidation of m the hydrolysis of Be and the presence of metals are discussed. Methods for preparin soln. suitable for the above procedure are desc for urine, bone, ores, steel, air-dust and s samples. Beryllium can be extracted with ac acetone and $CHCl_3$; heavy metals are compl with EDTA. P. D. PARR-RICHA

4292. **Rapid determination of beryllium in mineral.** J. L. Huguet and C. L. Bamb (Comisión Nacional de Energía Atómica, Bu Aires). Second U.N. International Conferenc the Peaceful Uses of Atomic Energy, Geneva, Rep. A/CONF.15/P/1578, 7 pp.—A study was m of the attack of mineral beryl with Na_2CO_3 a the determination of Be by the direct pptn. duced by the attack, in which no previous se tion (including that of SiO_2) has been made. conclusion is reached that, provided that precautions are observed, the technique ren applicable, markedly reducing the time us taken by the conventional method.

NUCL. SCI. ABS

4293. **Determination of the oxygen conte beryllium metal by activation.** R. F. Coleman J. L. Perkin (U.K.A.E.A., Atomic Weapons Estab., Aldermaston, Berks., England). *An* 1959, **84**, 233–236.—The beryllium sample irradiated in a 14·5-MeV neutron flux and the dual activity was measured with a Geiger cou The 7·4-sec. β-activity from ^{16}N produced in reaction $^{16}O(n,p)^{16}N$ was then compared wit activity from a similar-sized sample containi known amount of O. The neutron flux was tained by bombarding a tritiated zirconium t with 500-keV deuterons, the output being n tained at $\simeq 10^{10}$ neutrons per sec. with an expo of the sample of 1 to 10 sec. For the calibrati the standard beryllium, known mixtures beryllium and its oxide were prepared in an a sphere of argon. These were compacted sintered *in vacuo* and machined to a standard corrections for variation in neutron dose, cou geometry and self-absorption of β-particles thus avoided. The added O in the five sam used ranged in equal increments from nil to 1·(Each was irradiated and counted as descr The method had satisfactory precision with amo of O down to 0·001%. Five impurities with lives of 5 to 15 sec. could occur in beryllium, S, Si, Al, Mg and F. The max. limits for thes given. As little as 0·005% of O can be meas with an error of ± 20%. A. O. JON

4294. **Azo-dye derivatives of chromotropic salicylic acids as reagents for magnesium.** I Korenman and E. I. Levina. *Uch. Zap. Gor'k Univ.*, 1958, (32), 149–160; *Ref. Zhur., Kh* 1959, (1), Abstr. No. 919.—The reactions of a number of derivatives of chromotropic and sali acids with the alkaline-earth elements and I alkaline and ammoniacal media are studied. of the salicylic acid derivatives do not give chara istic colour reactions with these cations. The

azo derivatives of chromotropic acid with IO_2 (**I**) and $-(C_6H_4)_2{\cdot}N:N{\cdot}C_6H_3(OH)_2(SO_3H)_2$ hich can be used to detect 0·5 to 1·0 μg of a limiting dilution of 1 in 2×10^6 in the e of 300-fold amounts of alkaline-earth To 1 ml of soln. add 1 or 2 drops of a the appropriate reagent and 3 or 4 drops of H or 4 *N* aq. NH_3, mix and warm to between l 40° on a water bath. In the presence of colour of **I** changes from pink to blue or NH_3) dark lilac, and the colour of **II** from ink - lilac in aq. NH_3) to blue. **I** and **II** are st sensitive reagents for Mg yet known.

C. D. KOPKIN

Determination of magnesium in slags and by flame spectrophotometry. J. Coulombeau Maire (I.R.S.I.D., Saint-Germain-en-Laye,). *Chim. Anal.*, 1959, **41** (1), 3–11.—Con- are defined for the spectrophotometry of the ium line at 2852·1 A. The method requires ıoval of SiO_2 but not of other oxides. The soln. (*A*) is prepared by dissolving 2 g with vaporating twice to dryness with HCl to SiO_2 insoluble, taking up with HCl and filter- he residue is evaporated with HF to remove ısed with $NaKCO_3$ or $KHSO_4$, taken up with dded to the original filtrate, and the whole ıp to 500 ml. Working soln. are prepared as . For Thomas slag containing 2 to 6% of ke 10 ml of *A*, and make up to 100 ml with a *B*) containing 60 ml of conc. $HClO_4$ and 34 ml . HCl per litre. For Thomas slag containing 2% of Mg, take 10 ml of *A*, 10 ml of a soln. ıtaining 0·418 g of $MgCl_2.2H_2O$ per litre, and ıp to 100 ml with *B*. For substances other homas slag, take 10 ml of *A*, 10 ml of a soln. ntaining 1 ml of conc. H_3PO_4 and 3·68 g of H_2O per litre, and make up to 100 ml with r substances containing little Mg, take 10 ml f *A*, *D* and *C*, and make up to 100 ml with *B*. ıanipulation of the Beckman DU spectro- ıeter (equipped for flame) is described. The ıce between the peaks at 2843 and 2849 to is measured, six soln. and two standards used in a specified manner, and the result ed by linear interpolation; MgO (%) = $\times$ 0·0415, where *n* is the observed result in and *d* is the dilution. The actual measure- of six soln. occupies 1 hr., and the claimed cy is within $\pm$0·2% of the absolute value.

R. E. ESSERY

. Simple spectrophotometric method for deter- magnesium, calcium, strontium, barium, ım and zinc with ethylenediaminetetra-acetic J. R. Dunstone and E. Payne (Biochem. Univ. of Queensland, Brisbane, Australia). *t*, 1959, **84**, 110–113.—To construct the tion graph, soln. containing 1·0 to 5·0 s of metal ion are added to 1 ml of 0·005 *M* (disodium salt) and 1 ml of a buffer soln. l - aq. NH_3, pH 10). Each mixture is l to 5 ml and its extinction at 225 mμ is red against a blank soln. of 1 ml of buffer diluted to 5 ml. The differences between tinctions of these soln. and that of a mixture l of EDTA soln., 1 ml of buffer soln. and 3 ml er are plotted against the concn. of metal ion. l aliquot of the sample soln. containing 1 to les of the metal ion is treated in the same way, he extinction difference is referred to the tion graph. The method is useful when an cy within $\pm$3% is acceptable. In addition metal ion being determined, soln. for analysis may contain only the alkali-metal ions (except Li) and NH_4^+. The method fails if anions absorbing strongly at 225 mμ (*e.g.*, NO_3^- and acetate) are present, but PO_4^{3-} in concn. 40 times that of the metal ion can be tolerated in the determination of Ca.

A. O. JONES

4297. **Micro-determination of calcium.** A. B. Gilbert (King's College, Univ. of Durham, Newcastle upon Tyne, England). *Nature*, 1959, **183**, 888.—Concentrations of Ca of $\simeq 10^{-2}$ μg have been determined in vol. of $\simeq$40 μl of soln. with an accuracy of 1%, by using as indicator Na 1-(1-hydroxy-2-naphthylazo) - 5 - nitro - 2 - naphthol - 4 - sulphonate which is as suitable as but cheaper than Calcon (C.I. Mordant Black 17). The method is essentially similar to that of Hildebrand and Reilley (*Anal. Chem.*, 1957, **29**, 258).

K. A. PROCTOR

4298. **Improved end-point by addition of poly(vinyl alcohol) in the EDTA titration of calcium with Calcon as indicator.** P. F. Lott and K. L. Cheng (Dept. of Chem. Engng, School of Mines and Metall., Univ. of Missouri, Rolla, U.S.A.). *Chemist Analyst*, 1959, **48** (1), 13.—The earlier procedure (*cf. Anal. Abstr.*, 1957, **4**, 3576) for the stepwise determination of Ca and Mg with EDTA has been improved by the substitution of poly(vinyl alcohol) (**I**) for gelatin to inhibit the adsorption of Calcon on the pptd. $Mg(OH)_2$ in the first stage. A soln. of **I** is prepared by mixing 2 g of a medium-viscosity **I** with 200 ml of boiling water in a homogeniser. The soln. keeps indefinitely. To the test soln. (up to 0·5 millimole of Ca plus Mg) add 2 or 3 drops of the soln. of **I**, adjust the pH to $>$12·5 with NaOH, add 3 drops of 10% KCN soln. and 3 drops of Calcon (C.I. Mordant Black 17) indicator soln. and titrate with 0·01 *M* EDTA to a red to blue end-point. (Soln. high in Mg should be warmed to 60° before adding the indicator, and titrated hot.) Then bring the pH to 10 with ammoniacal buffer, boil to dissolve $Mg(OH)_2$, add 1 ml of buffer and 1 drop of Eriochrome black T soln. and titrate hot to a red to blue end-point with EDTA soln. Heavy-metal interference is dealt with as in the original method.

R. E. ESSERY

4299. **Partition of metal - 8-hydroxyquinoline compounds between water and organic solvents. IV. Investigation of the separation of calcium oxinate complexes and their photometric determination in chloroform solution.** F. Umland and K.-U. Meckenstock (Inst. f. Anorg. Chem., Tech. Hochschule, Hanover, Germany). *Z. anal. Chem.*, 1959, **165** (3), 161–179.—Calcium forms several complexes with 8-hydroxyquinoline (**I**). The normal Ca - $\mathbf{I}_2$ complex is extracted at pH 11·3 with $CHCl_3$ soln. containing $\not>$ 7% of **I**, but if the $CHCl_3$ soln. of **I** contains 2% of *n*-butylamine, Ca is extracted quant. at pH 11 to 12 as $C_4H_9NH_3[Ca - I_3]$ which is used for the photometric determination of Ca by measuring the extinction of the organic layer at 370 to 380 mμ. The average error is $\pm$ 2% for 0·8 to 9·6 μg of Ca per ml and $\pm$10% for 0·16 to 0·64 μg of Ca per ml. Interfering cations are either complexed with KCN or removed by a pre-extraction without the addition of butylamine. Detailed procedures are given and the practical application of the method is demonstrated.

B. B. BAUMINGER

4300. **Rapid determination of calcium oxide in flux agglomerate, limestone, dolomite, and blast-furnace and open-hearth slag, in the presence of triethanolamine as complexing agent.** V. D.

Konkin. *Byull. Nauch.-Tekhn. Inform. Ukr. Nauch.-Issled, Inst. Metall.*, 1958, (6), 111–115; *Ref. Zhur., Khim.*, 1959, (3), Abstr. No. 7978.—To remove the interference due to Al, Cr, Mn and Mg in the complexometric determination of CaO, these metals are masked by means of triethanolamine (**I**), which form complexes with them that are more stable than those with EDTA (disodium salt) (**II**). *Procedure*—Dissolve 0·1 g of finely ground agglomerate by warming with 7 ml of conc. HCl, cool, add water (90 ml), indigo carmine soln. (0·25 g in 100 ml of 50% ethanol) (5 drops) and **I** (1·5 ml). Shake vigorously for about 30 sec., add 12 ml of 20% NaOH soln. and 0·15 g of a mixture of murexide and NaCl (1:100), mix well and titrate with a soln. of **II** (≃0·5%). Determine the concn. of the **II** soln. with a soln. of CaO and with a standard flux agglomerate. For difficultly soluble agglomerates, mix the sample with an equal amount of Na_2CO_3, heat for 2 min. at 850° or 900°, cool the sintered mass, dissolve it in HCl and continue as described above. To determine CaO in blast-furnace and open-hearth slags, first shake the sample (0·1 g) vigorously with 60 ml of boiling water, immediately add 20 ml of conc. HCl and continue shaking for a further 1 or 2 min. to dissolve Ca completely. The presence of ⩾ 80 mg of Fe, 20 mg of Al, 8 mg of Mn, 20 mg of Mg and 4 mg of Cr does not cause interference. The determination of CaO in agglomerates takes about 15 min., and in limestone and slags 8 to 10 min. C. D. KOPKIN

4301. **Determination of zinc in aluminium and copper alloys.** M. Freegarde (Bragg Lab., Naval Ordnance Inspection Dept., Sheffield, England). *Metallurgia, Manchr*, 1958, **58**, 261–262.—Zinc (and some Cu) is adsorbed from 0·12 *N* HCl, containing 10% of NaCl, on the strongly basic anion-exchange resin De-Acidite FF. Copper is eluted with 2 *N* HCl and Zn is then eluted with *N* HNO_3 and titrated with EDTA. Magnesium, Fe, Mn, Ni, Ti and Si do not interfere, but Sn must be removed. The method is satisfactory for the determination of Zn in the range 0·1 to 6% in alloys of copper and aluminium, and its accuracy is equal to that of classical methods.
J. W. O. PYEMONT

4302. **Simple apparatus for the determination of mercury by amalgamation with gold.** J. Lacy (African Explosives & Chem. Industries Ltd., P.O. Northrand, Transvaal). *Anal. Chim. Acta*, 1959, **20** (2), 195–197.—The apparatus is intended particularly for the determination of Hg in charcoal activated with $HgCl_2$. It consists of a 15-ml silica flask surmounted by a silica column that contains 5 to 6 g of crumpled gold foil ≃0·0025 cm thick. A sample (1 g), containing ⩾ 70 mg of Hg, is mixed in the flask with 1 g of fine iron powder and the mixture is covered with a 0·3-cm layer of ZnO. The weighed column is inserted in the neck of the flask, which is then heated with a low flame for 10 min. and at a dull red heat for 20 min., the column being protected with a shield from the heat of the burner. After cooling, the column is rinsed with acetone, dried and weighed. The column is regenerated by heating in a bunsen flame while air is passed through it to remove the Hg.
H. N. S.

4303. **Solubility and amphoteric properties of mercuric oxide.** T. M. Salem (Chem. Dept., Univ. Coll. for Girls, Ain Shams Univ., Heliopolis, Cairo, Egypt). *J. Indian Chem. Soc.*, 1959, **36** (2), 83–86.—Mercury is determined potentiometrically wi error of ⩾ 1% for concn. as low as 0·5 mg per 10 Both $HgNO_3$ and $Hg(NO_3)_2$ soln. are titrated standard KI soln.; an amalgamated plat electrode (1 sq. cm) acts as indicator electrod is connected to a calomel reference electrode KNO_3 - agar bridge. The method is applied t determination of the solubility of HgO in and in soln. of various pH values. At 35° solubility is $3{\cdot}47 \times 10^{-4}$ moles of Hg per litre the iso-electric point occurs between pH 7·5 8·0. J. H. WAT

4304. **Spectrographic determination of boro aluminium.** B. Weszprémy and T. Török (Iron and Metal Works, Budapest). *Acta Acad. Sci. Hung.*, 1959, **19** (4), 357–361 (in Ger —The method is applicable to concn. of B us aluminium alloys, namely 0·01 to 0·5%. Stan for use in the routine procedure were pre from boron and aluminium of 99·99% purity analysed by spark spectroscopy in soln., the pair B 2497·72 - Al 2567·98 being used. routine analysis is carried out by a low-vo technique with solid electrodes. The line B 2497·72 - Al 2459·82 is used. The interf effect of Fe is corrected from an intensity mea ment of the line Fe 2486·35. The coeff. of vari of 12 determinations was ± 5·7%. G. BURG

4305. **Accurate determination of micro amounts of boron in aluminium and alumini uranium alloys by the methyl borate - curcu oxalic acid method.** I. H. Crocker (Atomic E of Canada Ltd., Chalk River, Ontario). *A Energy of Canada Ltd.*, Rep. CRDC–811, 26 pp.—The method is suitable for B conc 30 p.p.m. or higher. Boron is separated by dis tion as methyl borate from a HCl soln. of the and is determined spectrophotometrically by boric acid - curcumin - oxalic acid colour rea A precision of ±2% is attainable when the d mination is performed with the utmost care. accuracy is such that no bias need be given w calibration curve is used. NUCL. SCI. ABS

4306. **Determination of boric acid in glas distillation of methyl borate with circulatio methanol.** P. Ehrlich and T. Keil (Anorg. Chem. Inst., Univ., Giessen, Germany). *Z. Chem.*, 1959, **165** (3), 188–193.—The principle c circulation system, which has been used by Pi and Ehrlich (*Angew. Chem.*, 1953, **65**, 131) fo determination of fluorine, has been adopted fo distillation of methyl borate. The apparat illustrated and the procedure for the distill described. The distillate passes to a quartz from which the methanol is boiled off and re to the reaction flask. The alkaline soln. is ne ised with HCl, with an indicator of pH 4·4 t In the volumetric determination, mannit added and the boric acid is titrated using α- tholphthalein as indicator. Smaller amount boric acid are determined colorimetrically by method of Hatcher and Wilcox (*cf. Brit. Abs* 1950, 437), which is based on the reaction of B a soln. of carmine in conc. H_2SO_4. The av error in the volumetric method is <3% for 10 mg of B and in the photometric method < for 1 to 20 μg of B. The practical applicati the method is demonstrated by the determin of B in prepared mixtures of borax and silica a technical glass samples. B. B. BAUMING

Qualitative testing for borates by poly(vinyl . A. J. Monte-Bovi, J. J. Sciarra and C. ana (St. John's Univ., Coll. of Pharm., a, N.Y., U.S.A.). *Drug Standards*, 1959, 15–17.—The blue colour given by borates oly(vinyl alcohol) and iodine is a more e test than the colour with turmeric paper green flame produced on ignition with meth- nd H_2SO_4. A borate - iodine reagent is ed for the detection of poly(vinyl alcohol).

A. R. Rogers

Polarographic determination of aluminium. Bezuglyĭ (Kharkov Dental Materials Works). *Lab.*, 1959, **25** (3), 277–280.—The method is on the use of Ca gluconate (*cf.* Pecsok *et al.*, *r. Chem. Soc.*, 1955, **77**, 202, 1489). With a oln. containing 5% of Ca gluconate and 3 to $CaCl_2$, $E_{\frac{1}{2}}$ for Al is $-1{\cdot}57$ to $-1{\cdot}60$ V *vs.* .E.; the wave is well defined and the relation n wave height and concn. is linear.

G. S. Smith

Solvent extraction of aluminium oxinate. to Kambara and Hiroshi Hashitani (Dept. of Ritsumeikan Univ., Kyoto, Japan). *Anal.* 1959, **31** (4, Part I), 567–569.—A theory extraction of Al oxinate by organic solvents veloped, and confirmed experimentally. (8-hydroxyquinoline) (**I**) exhibits amphoteric ies, and its distribution as cationic, anionic ol. species in $CHCl_3$ or in H_2O is deduced; an ion is also given for the extractability of Al . The method of "logarithmic plot" used analysis of polarographic current - voltage is applied to find pH values corresponding to traction or half-dissociation. The pptn. of from neutral soln. can be avoided by **I** to the soln. before pH adjustment, followed raction with $CHCl_3$.

P. D. Parr-Richard

Determination of aluminium in the rare Yu. A. Chernikhov and B. M. Dobkina Sci. Res. Inst. of Rare Metals). *Zavod.* 1959, **25** (2), 131–132.—Extraction of the f the rare earths at pH 5 with $CHCl_3$ con- oxine separates Al which is then determined ietrically in the extract. With neodymium - sium alloys the sample (0·1 g) is attacked first cold and then by gentle heating with 3 ml . HCl. With chloride melts obtained in the ent of loparite ores the sample (0·1 g) is ed in water containing 0·5 ml of conc. HCl. ner case the soln. is evaporated nearly to s and the residue is dissolved by heating in 12 ml of water containing three drops of acid. The soln. in a separating-funnel is with 0·1 g of hydroxyammonium chloride ain Ce in the Ce^{3+} form) and several ml of an buffer soln. to pH $\simeq$ 4·5, and then with ml of $CHCl_3$ and 0·5 ml of 3% Na diethyl- arbamate soln., and shaken. The separated er is again extracted with $CHCl_3$ with the n of 0·2 ml of the thiocarbamate soln., and HCl_3 soln. containing Cu, Fe and Ni are d. If a further extraction gives a coloured he extraction is repeated. The aq. soln. is with 2 ml of 2 *M* Na acetate (the pH should $\simeq$ 5) and, if Th is present, with 4 to 5 ml of K H phthalate, and then shaken with a 0·5% f oxine in $CHCl_3$ (once with 2 ml and once ml). The Al in the extract is determined y by comparison with standards or by colori- metric titration, or photometrically at 380 to 400 mμ. The method is sensitive to 0·01% of Al.

G. S. Smith

4311. **Potentiometric determination of aluminium in nickel-based heat-resisting alloys.** A. A. Gorelova and L. Ya. Polyak. *Zavod. Lab.*, 1959, **25** (3), 285–287.—The determination of Al by titration with NaF soln. can be carried out either at pH 6 in the presence of a large amount of NaCl (whereby difficultly soluble cryolite is formed) or at pH 3·5 to 4·5 (whereby soluble, practically undissociated, AlF_3 is formed). To determine Al in alloys of Ni, Cr, Fe, Mo, W and Ti, the sample (0·25 g) containing 3 to 6% of Al is dissolved in 30 ml of aqua regia, and the soln. is evaporated to dryness, followed by treatment with 15 ml of dil. HCl (1:1) and re-evaporation. The residue is dissolved in 15 ml of dil. HCl (1:1) and 35 ml of water, and Cu, As, Sn and Sb are pptd. whilst Fe, Ti and Mo are reduced by treatment of the soln. with granulated zinc. After 5 min. the remaining zinc is filtered off and washed with 1% HCl soln. (3 × 10 ml) and then with water, and the filtrate and washings are neutralised with aq. NH_3 and dil. HCl. For the cryolite method, the soln. (100 ml) is treated with 3 ml of dil. HCl (1:1), 10 g of NaCl and 25 ml of 50% Na acetate soln. to give a pH of 6, and then titrated with 0·25 *N* NaF to the first sharp potential jump. The indicator electrode is of aluminium and the reference electrode of nichrome, spaced 5 cm apart and immersed to a depth of 1 to 2 cm. For the AlF_3 method, the soln. (100 ml) is treated with 3 ml of dil. HCl (1:1) and 10 ml of 50% Na acetate soln. to give a pH of 3·5, and titrated. The methods are equally accurate.

G. S. Smith

4312. **Photometric determination of small amounts of phosphorus in aluminium alloys.** Z. Večeřa and B. Bieber (State Res. Inst. of Materials and Technol., Brno, Czechoslovakia). *Hutn. Listy*, 1959, **14** (1), 56–58.—*Procedure*—To the sample (1 g) in a distillation flask add H_2O (5 ml), remove air by bubbling for 10 min. with CO_2, add HCl (1:1) (25 ml) and distil the PH_3 into a vessel containing Br, with occasional addition of HCl. Remove the excess of Br by heating, add a small amount of Na_2SO_3, boil for 3 min., transfer to a 100-ml separating-funnel, dilute with H_2O to 40 ml, add *N* H_2SO_4 (2 ml), ammonium molybdate soln. (100 ml of 5% ammonium molybdate soln. mixed with 100 ml of 10 *N* H_2SO_4) (5 ml) and extract with 40 to 50 ml of butanol. Wash the butanol layer with 0·5 *N* H_2SO_4 (2 × 40 ml), transfer it to another separating-funnel containing 40 ml of *N* H_2SO_4 and $SnCl_2$ soln. (0·5 ml), shake for 2 to 3 min., separate the aq. layer, dilute the blue butanol layer to 100 ml with butanol and measure the extinction at 720 mμ after 20 min. Compare with a calibration curve. Silicon hydride and H_2S do not interfere. Interference by As can be avoided by the addition of H_2SO_3 or thiourea. The method requires $\geq$ 1·5 to 2 hr. and has been tested with good results by the analysis of silumin samples containing 10% of Si, 0·2 to 0·4% of Mn, 0·2% of Fe and 0·3% of Mg.

J. Zýka

4313. **Photometric determination of gallium using Malachite green.** J. Jankovský (Inst. for Ore Res., Prague). *Talanta*, 1959, **2** (1), 29–37.—Malachite green (C.I. Basic Green 4) forms a blue - green benzene-soluble complex with Ga in conc. HCl soln. *Procedure*—Transfer a 5-ml aliquot containing up to 5 μg of Ga in HCl (1:1) to a dry

25-ml flask, add conc. HCl (1 ml), $TiCl_3$ soln. (15%) (0·5 ml) and mix. After 5 min. add Malachite green soln. [2% in HCl (1:3)] (2 ml). Mix, add 5 ml of benzene and shake for 2 min. Measure the extinction of the benzene layer at 660 mμ in a 1-cm cell. The method is suitable for the determination of 0·001% of Ga in bauxite, zinc ores and ashes; smaller quantities can be determined after a concentration step comprising extraction with di*iso*propyl ether. The interfering effects of 64 foreign ions are tabulated. W. T. CARTER

4314. **Photometric determination of traces of gallium with stilbazo as reagent. I.** M. Z. Yampol'skiĭ. *Ich. Zap. Kursk. Gos. Ped. Inst.*, 1958, (7), 67–72; *Ref. Zhur., Khim.*, 1959, (2), Abstr. No. 4306.—Stilbazo forms with Ga^{3+} a red - violet compound. The intensity of the colour is proportional to the concn. of Ga up to 7 μg in 3 ml of soln., and increases with increase of pH, the optimum pH being 6·1 to 6·5. For the photometric determination of Ga, to 2 ml of ammoniacal acetate buffer soln. (pH 6·5) add the test soln. and 0·5 ml of a 0·001% soln. of stilbazo (total vol. 3·0 ml), and measure the extinction of the resulting soln. in a 5-mm cell at 530 mμ, with water as comparison soln. At pH 4·0 no interference is caused in the determination of <5 μg of Ga by the presence of >400 μg of Cd, >300 μg of Zn, and >200 μg of Al. The method may be used for the determination of Ga in metallic zinc; dissolve 1 g of technical zinc in 20 ml of HCl and, after electrolytic removal of Cu and Pb, determine Ga in the soln. by the method of additions (add 8 μg of Ga to 0·5 g of Zn). One determination takes 2 to 3 hr. C. D. KOPKIN

4315. **Investigation of the extractive separation of gallium and indium with different solvents.** H. Hartkamp and H. Specker (Inst. f. Spectrochem., Dortmund-Aplerbeck, Germany). *Talanta*, 1959, **2** (1), 67–72 (in German).—The procedure previously described (*Anal. Abstr.*, 1957, **4**, 1160) has been further studied. It is concluded that the In is best extracted with *cyclo*hexanone from a 0·2 *N* H_2SO_4 soln. containing a 6 to 10-fold excess of KI. Chloride and bromide must be absent. By the same procedure, In can also be quant. separated from Fe, Al and the alkaline-earth metals.
W. T. CARTER

4316. **New fluorimetric method for determining indium.** I. A. Blyum and T. K. Dushina (Central Ores Lab., Chelyabinsk Geological Trust). *Zavod. Lab.*, 1959, **25** (2), 137–139.—The method is based on the fluorescence excited by u.v. irradiation of benzene soln. of the complex formed b In andy Rhodamine 3B (C.I. Basic Violet 11) in the presence of HBr. By extracting 10 ml of a soln. of In, 2·5 *M* in HBr, mixed with 0·1 ml of 0·25% Rhodamine 3B soln., with 5 ml of benzene, and irradiating the extract, 0·02 μg of In can be detected. No interference is caused by K, Na, Mg, Ca, Cd, Al, Ga, Sn^{2+}, Sn^{4+}, Ti^{4+}, Ge, Sb^{3+}, Bi, V^{4+}, Cr^{3+}, Tl^{+}, W, Pt, Mo, Co, Ni, Fe^{2+}, Mn^{2+}, Cu^{2+}, SO_4^{2-} and PO_4^{3-}, but fluorescence occurs with Au^{3+}, Tl^{3+}, Hg^{2+}, Sb^{5+}, Cu^{+}, Pb, V^{5+}, Cr^{6+}, Se^{4+}, Te^{4+}, As^{5+}, Fe^{3+}, Zn, Ag and NO_3^-. The reagent is most sensitive to In; thus the same fluorescence intensity is given by 1 μg of In as by 5 μg of Au, 4 μg of Tl, 5 μg of Te, 8 μg of Hg, 20 μg of Ag, 40 μg of $Cr_2O_7^{2-}$, 2000 μg of Pb or 6000 μg of Zn. By preliminary reduction with iron, the interference of Au, Ag, Tl, As, Se, Te, Fe, Cr, Sb and V can be prevented. For general use in determining In, a preliminary extraction of $InBr_3$ with butyl a is recommended. To determine In in ores and Zn, etc., the sample (0·1 g) is dissolv treatment in the cold with 10 ml of conc. and 5 ml of conc. HCl followed by gentle h for 2 to 3 hr. The soln. is evaporated nea dryness and the residue is mixed with 3 t of conc. HBr and the soln. is again evapo the treatment with HBr and evaporation repeated. The dry residue is dissolved in of 5 *N* HBr and the In is extracted with 1 butyl acetate. The extract is washed wit HBr (2 × 2 to 3 ml) and the In is re-ext with dil. HCl (2:1) (2 × 10 ml). After the ad of 1 mg of $FeCl_3$ or $Fe_2(SO_4)_3$ (as a collecto 5 ml of dil. H_2SO_4 (1:1) and 1 ml of H_2O_2 so destroy organic matter) the soln. is evapora fuming, a further amount of H_2O_2 being ad the residue chars. The residue is dilut between 30 and 40 ml and treated with an ex aq. NH_3. The collected and washed ppt. is dis in 2·5 *N* HBr and the soln. is treated with 0 0·03 g of iron (reduced with H) and set aside hr. The filtered soln. is treated with two dr a 0·10 soln. of Rhodamine 3B in water a complex is extracted with 5 ml of benzene. fluorescence of the extract is compared with t standards. G. S. SM

4317. **Co-precipitation of thallium with sulphide.** G. V. Efremov and S. A. Leo *Vestn. Leningr. Univ.*, 1959, No. 4, *Ser. Khim.*, (1), 141–144.—The conditions for co-pptn. as regards the relative proportions and Zn, the pH of the soln. in which the pptn. place, the dilution of the soln. and times re for pptn. are specified. The data obtained in that this co-pptn. can be used for the analy zinc metal, zinc ores and zinc waste, and f micro-determination of Tl. K. R

4318. **Photometric determination of total earth elements in ores and rocks.** F. V. Zaĭk and V. S. Bashmakova (All-Union Inst. of Raw Materials, Moscow). *Zhur. Anal.* 1959, **14** (1), 50–54.—It is established that s the complexes of arsenazo with rare-earth ele of the cerium and yttrium groups obey Beer' To determine total rare-earth elements, deco the sample (0·025 to 0·25 g) by fusing with extract with hot water, filter off and was hydroxides, dissolve them in hot 5% HCl 30 ml), add saturated Na acetate soln. dropw pH 5 (Congo red) and dilute to 100 ml. Ad $CaCl_2$ soln. (0·5 ml) and 15 ml of a soln. of dic acetone (≡ 4 g of oxalate ion), boil for 15 set aside overnight, and filter and wash th with 1% oxalic acid soln. Ignite the ppt. at evaporate the residue with 3 to 4 ml of conc dissolve the chlorides (of the rare-earth ele Th and Ca) in water (30 ml), neutralise to red with 10% aq. NH_3, add HCl (sp.gr. (0·5 ml) and a 0·1% soln. of thoron, stir, add of activated charcoal, stir and filter. Repe charcoal treatment; this removes Th and tra Zr. To the filtrate add aq. NH_3 to pH 4, and up to 50 ml. To an aliquot of this soln. (cont 5 to 70 μg of total rare-earth elements) ad ascorbic acid soln. (0·5 ml), 10% sulphosa acid soln. (0·5 ml), 0·05% arsenazo soln. (3 m 20% hexamine soln. (4 ml), make up to 25 m after 30 min. measure the extinction at 57 A blank is prepared from an equal aliquot final soln., with the same additions except

l of a 10% tartaric acid soln. replaces the salicylic acid soln. The calibration curve is ucted from 25 ml of soln. containing 5 to 70 otal rare-earth elements, 5 μg of Ca, and the reagents used in the analytical soln. described the blank contains all reagents but no rare-elements. The method permits the deter-on of 0·01 to 3% of rare-earth elements with of 5 to 20%. C. D. KOPKIN

9. **Thermal decomposition of the rare-earth-oxalates.** W. W. Wendlandt (Dept. of Chem. hem. Engng, Texas Technol. Coll., Lubbock, .). *Anal. Chem.*, 1959, **31** (3), 408–410.—The es of Tb, Dy, Tm, Yb and Lu were prepared 10-, 6- or 5-hydrates by homogeneous pptn. nethyl oxalate, and 70 to 90-mg samples were l at 5·4° per min. on a thermobalance, in a tream of air; Tb and Dy form unstable inter-te hydrates; stable hydrate weight levels btained for Tm, Yb and Lu. The dihydrates urprisingly stable, but the anhyd. oxalates pose rapidly, liberating CO and CO_2. Losses ght of the hydrates begin at 45° to 60°, and ides are finally obtained at 715° to 745°.
P. D. PARR-RICHARD

0. **Separation of rare-earth elements from um, magnesium, zirconium, titanium, uranium ainless steel.** M. W. Lerner and L. J. Pinto Brunswick Lab., U.S. Atomic Energy Comm., *Anal. Chem.*, 1959, **31** (4, Part I), 549–551.— earth elements present in metals to be used uclear technology must be isolated prior to spectrographic determination, and detailed dures are given for the separations cited .
P. D. PARR-RICHARD

1. **Separation of iron from rare-earth elements.** Křepelka, K. Vetejška and J. Mazáček (Res. Ores, Prague). *Coll. Czech. Chem. Commun.*, **24** (1), 198–202.—The separation of Fe from arth metals on the strongly basic ion-exchange OAL has been investigated. The rare-earth nts and Th are not adsorbed from an HCl m and can be determined in the percolate, as Fe is retained on the column and can be and determined in the usual way. Zir-n is also retained on the column, but does not ere. The procedure has been tested by analys-nonazite concentrates. The sample is fused an excess of Na_2O_2, washed with hot H_2O till al (removal of sodium phosphate), then ved in HNO_3. Iron, together with rare-metals, Th, Zr and Ni, is pptd. with aq. NH_3, pt. is washed with hot H_2O and dissolved on lter with hot HCl. The soln. is then added OAL column previously washed with 8 *N* Fe is eluted with hot H_2O and determined etrically. Rare-earth elements and Th can termined gravimetrically after pptn. with aq. or oxalic acid. J. ZÝKA

2. **Ion-exchange separation of cerium and m with lead - EDTA complex as eluting agent.** tsugu Taketatsu (Chem. Dept., Fac. of Sci., nu Univ., Hakozaki, Fukuoka). *Bull. Chem. Japan*, 1959, **32** (3), 291–293 (in English).—optimum conditions for this separation on x-50 resin (NH_4^+ form) are a concn. of the EDTA soln. of 0·015 *M* at a pH of 3·0 and a rate of 1·0 to 1·5 ml per min. Thorium ions ompletely eluted with 200 ml of the eluent under these conditions, while no cerium ions are detected in the eluate even after using 1 litre of the eluent. After elution of Th, the column is washed with water, and Ce is eluted with 3 *N* HCl.
I. JONES

4323. **Spectrographic determination of small quantities of lanthanum, praseodymium, neodymium and yttrium in cerium.** Pao-Lin Hsu, Lien-Fang Shen, Chien-Hwa Cheng and Ting-Chao Chang. *Acta Chim. Sinica*, 1958, **24** (6), 419–425.—Two methods for the determination of rare-earth-element impurities in "purified" cerium are described. (*i*) The sample is dissolved in HCl (1:19) so that the concn. of CeO_2 is 10 mg per ml. A drop of the prepared soln. is transferred to the flat top of a graphite electrode previously treated with liquid paraffin. The electrode is dried on a hot-plate and is then excited in an a.c. arc (220 V, 5 amp.). A spectrograph ISP-51 (with UF-85 auto-collimating camera tube) is used, with a slit width of 0·01 mm. By using the line pairs La 4333·74 - Ce 4330·90, Pr 4225·33 - Ce 4229·63, Nd 4061·09 - Ce 4057·30, and Y 4374·94 - Ce 4376·88, La, Pr, Nd and Y can be determined quant. in the concn. ranges 0·055 to 0·62%, 0·089 to 1·29%, 0·052 to 0·59% and 0·025 to 0·40%, respectively. The coeff. of variation for single determinations are $\pm$ 4·7%, $\pm$ 10·8%, $\pm$ 7% and $\pm$ 7%, respectively. (*ii*) For cerium samples of high purity, ether extraction is used to remove most of the Ce and concentrate the rare-earth-element impurities. Because of its relatively simple spectrum, the element Y has been used as carrier and internal standard. The concentrate, in HCl soln. (concn. 1 mg of rare-earth oxide per ml) is excited and photographed by the same method as in (*i*). The line pairs used are La 4238·38 - Y 4235·73, La 3988·52 - Y 3951·60, Pr 4222·98 - Y 4235·73, and Nd 4232·38 - Y 4235·73. With a sample of 100 mg (1 mg of Y_2O_3 added as internal standard) the analytical sensitivities are 0·01%, 0·025% and 0·028% for La, Pr and Nd, respectively. The experimental error is $\simeq \pm$ 10%.
SCI. ABSTR. CHINA

4324. **An anomalous change in the intensity of the samarium lines in the direct-current arc.** R. R. Shvangiradze. *Optika i Spektroskopiya*, 1958, **5** (1), 88–90; *Ref. Zhur., Khim.*, 1959, (2), Abstr. No. 4310.—Experimental data are given of an anomalous change in the intensity of the spectral lines of Sm. A mixture of rare-earth-metal oxides and powdered carbon (1:1) was placed in a crater in a carbon electrode and ignited in a d.c. arc at 5 amp. It was found that the changes in the density of the lines, taken every 15 sec., were similar for all the rare-earth elements studied, with the exception of Sm. When the mixture contained mainly Sm, with small admixtures (0·02 to 0·1%) of the other rare-earth elements, at the end of the ignition of the Sm there was a sharp increase in the intensities of the lines of all the admixtures. This was not observed when any other rare-earth element was the main constituent. With increase of the current, the intensity of the rare-earth-element lines increases. The intensity of the Sm lines increases only up to a current of 6 amp., remaining almost constant thereafter. The conclusion is drawn that the anomalous behaviour of Sm is not due to processes taking place in the vapour cloud of the arc, but depends on an abnormal effect in the transfer of matter from the solid to the gaseous state. In the anode crater the Sm undergoes chemical changes which are different from those

of the other rare-earth metals. The vapour pressure of the samarium compounds entering the arc cloud is higher than that of the rare-earth elements. These anomalies are not observed in an a.c. arc between copper electrodes. On the basis of the results obtained, conclusions are drawn regarding the practical spectrographic determination of Sm and rare-earth-element admixtures in samarium concentrate. C. D. KOPKIN

4325. **Gravimetric determination of silica in fluorine-containing beryllium oxides.** J. L. Huguet (Comisión Nacional de Energía Atómica, Buenos Aires). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/1580, 5 pp.—A study is made of the applicability of the Schernk and Ode method to the gravimetric determination of the SiO_2 content of technical-grade beryllium compounds which contain some fluoride by reason of their origin. It is found that the method gives good results in the range for which it has been tried ($\simeq$ 1% of SiO_2). NUCL. SCI. ABSTR.

4326. **Hydroxyanthraquinones as reagents for germanium.** I. M. Korenman, N. V. Kurina and E. A. Emelin. *Trudy po Khim. i Khim. Tekhnol.*, 1958, (1), 134–137; *Ref. Zhur., Khim.*, 1959, (1), Abstr. No. 933.—The colour reactions of GeO_3^{2-} with 1:2:4:5:6:8-hexa- (**I**), 1:2:4-tri- (**II**), 1:5-di- (**III**), 1:4-di- (**IV**) and 1:2:5:8-tetra- (**V**) hydroxyanthraquinone are studied. A 0·01% soln. of the reagent in 95% H_2SO_4 is used. In all cases a colour is formed (the limiting dilution and the detectable minimum of Ge, in μg per 0·1 ml, are given in brackets)—with **I**, violet (1 in 7×10^4, 1·4); with **II**, red (1 in 5×10^4, 2·0); with **III**, orange, (1 in $3{\cdot}5 \times 10^4$, 2·9); with **IV**, red (1 in 3×10^4, 3·3); and with **V**, violet (1 in $1{\cdot}4 \times 10^4$, 7·0); **I**, **II** and **V** are the most selective of the reagents studied. Only BO_3^{3-}, Al^{3+} and Tl^+ interfere in the detection of Ge, since these also react with hydroxyanthraquinones. **I** and **IV** fluoresce in the presence of Ge, pinkish-red and clear yellow, respectively, this being noticeable even in ordinary light. The sensitivity of the fluorescent soln. is the same as for the corresponding colour reactions, but the selectivity is somewhat higher (Ge can be detected by **I** in the presence of twice the amount of Al). **I** can also be used for the photometric determination of Ge, measuring the extinction at 610 mμ. C. D. KOPKIN

4327. **Determination of germanium using the constant temperature d.c. arc method.** M. Malinek (Metallurg. Inst., Czech. Acad. of Sci., Prague). *Appl. Spectroscopy*, 1959, **13** (1), 1–3.—Germanium, in a variety of matrices, at concn. from 0·01 to 1·0%, can be determined by means of a single working curve. The results for 16 replicate determinations on one sample gave a coeff. of variation of $\pm$ 11%. K. A. PROCTOR

4328. **Rapid method of determining germanium in coal ash.** M. A. Menkovskiĭ and A. N. Aleksandrova (I. V. Stalin Moscow Mining Inst.). *Zavod. Lab.*, 1959, **25** (2), 161.—The sample (0·2 to 0·5 g) is decomposed by heating with 10 ml of syrupy H_3PO_4 for 20 to 30 min.; 10 ml of 6 *N* HCl is added, and $GeCl_4$ is distilled off into 5 to 6 ml of water. Further distillation is carried out after the addition of 10 ml of 6 *N* HCl. The distillate is diluted to 50 ml and Ge is determined colorimetrically in an aliquot by means of phenylfluorone in the presence of gelatin (*cf.* Nazarenko *et al.*, *Anal. Abstr.*, 1958, **5**, 3292). G. S. SMITH

4329. **Spectrographic method for quanti determination of germanium in zinc ores a metallurgical intermediate products.** S. Witk (Inst. of Non-Ferrous Metall., Gliwice). *Anal., Warsaw*, 1959, **4** (1–2), 471–475.—By m of spectrographic analysis the natural produc compared with a synthetic sample, similarly tr and corrected (*e.g.*, by addition of Al_2O_3, Ag_2 As_2O_3) so as to be almost identical with the na product. To 100-g portions of the synt sample, an appropriate amount of GeO_2, diss in 10% KOH soln., was added, and after dryi 105° the prepared standard was thoroughly h genised for spectrographic examination. Two pairs were used, with Bi as internal stand Bi 2938·30 A and Ge 2709·626 A, and Bi 2938 and Ge 3039·064 A, the second being used wh was absent. Materials varying greatly in com tion and volatility were examined by the energy method. The curve for the line-pa 2897·98 A - Ge 2657·178 A was satisfactory compared with classical methods. It is concl that the accuracy of the determination of Ge (to 0·05%) by the former (pellet) method is $\pm$ and by the total energy method $\pm$ 15·0%. W. B. MIASKOW

4330. **Polarographic reduction of quadriv germanium in complexing organic acid solu** M. Lucco Borlera (Ist. di Chim. del Politec., To Italy). *Ric. Sci.*, 1959, **29** (1), 100–105.— polarographic reduction of Ge^{4+} is investi with salicylic, oxalic and tartaric acids as plexing agents. The optimum pH range for r tion is—HCl - salicylic acid, 7·5 to 9; HCl - acid, 7 to 9; HCl - tartaric acid, 8·2 to 9·5. values of the diffusion current constants obt in various media are listed and the optimu range is given over which the reduction analytical value. The best complexing ac salicylic, with a 0·001 *M* Ge^{4+} soln. L. ZAN

4331. **Determination of copper admixture metallic germanium by activation analysis.** Gottfried and J. V. Jakovlev (Res. Inst. Chem., Ústí Nad Labem, Czechoslovakia). *Prûmysl*, 1959, **9** (4), 179–182.—The samp irradiated in a nuclear reactor by means stream of neutrons (5×10^{11} to 2×10^{13} neu per sq. cm per sec.) for 48 hr. and then dissolve mixture of HCl and HNO_3. Arsenic, Cu, Zn, Te, Sb and Ce are added as carriers; Ge and A separated by distillation, Te by a stream of (reduction), Sb and rare-earth elements as oxides, Mo with benzoin α-oxime from a sol HCl and Cu by pptn. from a slightly ammon soln. with the same reagent. The quant. separ of Cu from Zn is achieved by further pptn. H_2S and finally as CuSCN, the activity of is then measured. J. ZÝ

4332. **Determination of small amounts of le rocks.** Z. A. Baskova (All-Union Scientific Geol. Inst., Leningrad). *Zhur. Anal. K* 1959, **14** (1), 75–80.—The concentration of P the following methods is described, the centage of Pb extracted being given—(*i*) extra with dithizone and $CHCl_3$ (91%); (*ii*) co-pptn. HgS (60 to 70%); (*iii*) co-pptn. with ZnS (8 90%); (*iv*) co-pptn. with $BaSO_4$ (76%); (*v*) ex tion of Pb diethyldithiocarbamate with alcohol and toluene (1:1) (97%); and (*vi*) distill of Pb in the presence of C *in vacuo* or in a strea N (80 to 100%). The percentage extractions

found radiochemically, the final determina- f Pb being made polarographically or colori- ally with dithizone. C. D. KOPKIN

. **Spectrographic analysis of lead- and zinc- loys.** N. A. Kiseleva. *Poligr. Proizvodstvo*, (6), 23–25; *Ref. Zhur., Khim.*, 1959, (1), No. 1001.—The spectra are excited in a ised spark discharge from an IG-3 generator. lectrodes are prepared from the analytical s and are 6 mm in diam. rounded to a hemi- . The analytical conditions are—capacity μF, self-induction 0·01 mH, spark gap 2 mm, t 0·8 amp., sparking 1 min., exposure 1 min. pectra are photographed in a medium spectro- with a slit width of 0·03 mm. Calibration are constructed on the co-ordinates $\triangle S$ *C*, from the lines (in A) Sb 2598 and Pb 2628, 29 and Pb 2446, and Cu 3274, Zn 3345, Ni r Al 3082 and Pb 3220. The analysis takes 50 When determining Al, Mg and Cu in zinc alloys he lines at Al 3082·1, Mg 2790·7, Cu 3247·5 n 3075·9 A, the condensed spark discharge he generator in a simple circuit was used ity 0·005 μF, self-induction 0, current ., sparking 30 sec., exposure 30 sec., slit 0·08 mm). Lead, Cu, Sb and Sn are deter- by exciting the spectra in an a.c. arc dis- with an interrupter and a threefold attenu- The electrodes are ground to a flat surface. rc gap is 2·5 mm, current 7 amp., ignition 1 exposure 1 to 3 min., and slit width 0·04 mm. nalytical lines (in A) are Cu 3247·5 and Zn , Pb 2833·1 and Zn 2712·5, Sb 2528·5 and Zn , Sn 3175·0 and Zn 2712·5. The analysis 50 min. The standards are prepared by of the pure metals. C. D. KOPKIN

. **Chromotropic acid derivatives as reagents for m.** I. M. Korenman, F. S. Frum and L. V. ova. *Uch. Zap. Gor'kovsk. Univ.*, 1958, (32), 7; *Ref. Zhur., Khim.*, 1959, (1), Abstr. No. The possible use of a number of chromotropic zo dyes as colour reagents for Ti^{4+} is studied. yes are the azo derivatives of chromotropic vith $HO(Cl)C_6H_3-$ (**I**), $HO_3S(OH)C_6H_3-$ or C_6H_4-. All the dyestuffs studied are water e, giving soln. which may be kept for a long without change. The addition to 1 ml of a f $TiOSO_4$ of 5 drops of a satd. soln. of Na e and 3 drops of a 0·1% soln. of a dyestuff es a coloured ppt. in the presence of large ts of Ti, or a coloured soln. with low concn. It is established photometrically that the ion of the coloured product requires 2 moles er g of Ti. The most sensitive reaction is with **I** (limiting dilution 1 in 1×10^6); no rence is caused by Mg, Ca, Pb or other ts. Small amounts of Ti should be deter- photometrically with **I** at 550 mμ in a cell. To 3 ml of weakly acid test soln. add acetate (3 ml) and a 0·2% soln. of **I** (3 drops). 0 to 60 μg of Ti in 3 ml, the intensity of the is proportional to the concn. of Ti. C. D. KOPKIN

. **Study of certain azo dyes as reagents for m.** F. S. Frum and E. P. Trushina. *Uch. Gor'kovsk. Univ.*, 1958, (32), 119–125; *Ref. Khim.*, 1959, (1), Abstr. No. 938.—The colour ns of Ti^{4+} with 10 azo dyes are studied. ost sensitive reagent is $O_2N(Cl)(OH)C_6H_2$·- $_{10}H_5(OH)SO_3Na$ (**I**) which with Ti forms a k complex of composition Ti:**I** = 1:2. The limiting dilution is 1 in 2×10^6. For the detection of Ti with **I**, to 1 ml of weakly acid test soln. add a saturated soln. of Na acetate (5 drops) and a 0·2% soln. of **I** (2 drops); no interference is caused by W, Be, La or Fe. In the presence of Th, Ce, Zr or Mo, the use of $HO_3S(NH_2)(OH)C_{10}H_4{\cdot}N:N{\cdot}(C_6H_4)_2{\cdot}N:$- $N{\cdot}C_6H_3(OH)COOH$ is recommended, and in the presence of Cr, UO_2^{2+}, Al, Zn or Ca, the reagent $HO_3S(NH_2)(OH)C_{10}H_4{\cdot}N:N{\cdot}C_6H_4{\cdot}N:N{\cdot}C_6H_3(OH)$- COOH. For the photometric determination of Ti, to 5 ml of test soln. add a 0·2% soln. of **I** (0·35 ml) and a satd. soln. of Na acetate (0·75 ml), and measure the extinction with a green filter. The attenuation of the colour of the soln. is proportional to the content of Ti with 5 to 50 μg of Ti in 5 ml. The photometric method also gives completely satisfactory results in the presence of Mg, Ca, Mn, Al and Fe. C. D. KOPKIN

4336. **Determination of titanium with EDTA.** D. H. Wilkins (General Electric Co., New York, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (2), 113–116.—The method of back-titration with Cu^{2+} (*cf.* Šír and Přibil, *Anal. Abstr.*, 1957, **4**, 822) fails through the pptn. of hydrated TiO_2. This pptn. is now prevented by the addition of H_2O_2. *Procedure*—To the soln. of ≃10 mg of Ti in HCl or HF add 10 ml of dil. H_2SO_4 (1 + 1) and evaporate to fuming. Cool to room temp., add 10 to 15 g of ice and 3 drops of H_2O_2 soln. (30 vol.). Add a 5 to 10-ml excess of standard EDTA soln., dilute to 100 ml, add NaOH soln. (50%) to produce a pH of 4 to 5, and 8 drops of 1-(2-pyridylazo)-2-naphthol indicator soln. (0·05% in ethanol). Titrate with a standard soln. of Cu^{2+} to a change from orange to orange - red. The max. error recorded is −0·08 mg. H. N. S.

4337. **Extraction of titanium thiocyanate with tri-*n*-octylphosphine oxide. Direct colorimetric determination in the organic phase.** J. P. Young and J. C. White (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 393–397.—Titanium thiocyanate is extracted from acid SO_4^{2-} and Cl^- soln. with tri-*n*-octylphosphine oxide in *cyclo*hexane and the absorption is measured at 432 mμ. Beer's law is followed up to 1·7 μg of Ti per ml, and the molar absorption coefficient is ≃41,000. Neither quadrivalent metal ions nor large quantities of Fe^{3+} or of U^{6+} interfere; Nb in threefold excess over the Ti gives a 5% error. Fluoride, PO_4^{3-} and oxalate do not interfere, but NO_3^- cause decomposition of the SCN^-. Recoveries are generally > 95% and the coefficient of variation is ≃2%. Twenty-five diverse ions were tested for interference. G. P. COOK

4338. **Rapid method of determining hydrogen in metallic titanium powder.** R. N. Rubinshteĭn and N. G. Mendlina. *Zavod. Lab.*, 1959, **25** (1), 34–36.—A simple apparatus for the vacuum extraction at 600° to 700° and determination of H is described. G. S. SMITH

4339. **Preparation and analysis of titanium - hydrogen standard samples.** M. J. Trzeciak (Battelle Memorial Inst. Defense Metals Information Center, Columbus, Ohio). *U.S. Atomic Energy Comm.*, Rep. DMIC-Memo-9, 1959, 14 pp.—The preparation of standard Ti - H samples for use in standardisation and the evaluation of analytical methods for gases in metals is given. The preparation of materials and the hydridation procedure are described, and the results of sample analyses are compared with those of other laboratories. NUCL. SCI. ABSTR.

4340. **Chemical and spectrochemical analysis of zirconium and Zircaloy.** G. W. Goward and R. M. Jacobs (Westinghouse Electric Corp. Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-BT-11, pp. 81–103.—The development and use of chemical and spectrochemical procedures at the Bettis Plant for the analysis of zirconium and Zircaloy alloys are described. In addition, the establishment of certain reference standards, some of the difficulties encountered in the analysis, and plans for resolving the difficulties are discussed.
NUCL. SCI. ABSTR.

4341. **Determination of zirconium in plutonium by ion exchange and spectrography.** R. Ko (Gen. Electric Co., Richland, Wash., U.S.A.). *Appl. Spectroscopy*, 1959, **13** (1), 10–11.—Plutonium can be separated from Zr by retaining it as the nitrate complex on Dowex-1 anion-exchange resin, and collecting the Zr in the percolate and wash soln.; Zr is then determined, after evaporation of the soln., by graphite spark excitation, with Co as the internal standard. Concn. as low as 20 p.p.m. of Zr have been determined with a precision of $\pm 17\%$ for a single determination. K. A. PROCTOR

4342. **Spectral analysis of impurities in zirconium and its compounds.** Y. I. Korovin and L. V. Lipis. Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/2137, 15 pp.—A study was made of a direct spectral method, with high sensitivity and reasonable accuracy, for the simultaneous determination of 24 elements in zirconium and its compounds. The method is based on the use of a discharge in a hollow cathode. The temp. of the sample, which is under constant discharge conditions, is unambiguously related to the discharge current. This makes it possible to considerably reduce the influence of the third components in determining a large group of elements owing to a more complete application of the fractional evaporation principle. With many elements it is possible to use direct density measurement owing to the high stability of the source of light, while in other instances the use of an internal standard considerably improves the calibration curves and the reproducibility of results. The method is quicker and more labour-saving than the conventional methods; it is possible to determine the impurities in zirconium with high sensitivity, and such determinations, although not optimum, meet the requirements for the quality of zirconium and its compounds. NUCL. SCI. ABSTR.

4343. **Spectrochemical determination of residual hafnium in zirconium and zirconium alloys using d.c. arc excitation and a barium fluoride flux.** G. J. Harter and R. F. Farrell (Westinghouse Electric Corp. Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-162-10, 1958, 6 pp.—This method is used to determine residual Hf in zirconium and Zircaloy from 30 p.p.m. up to 300 p.p.m. By selection of suitable line pairs the method may be extended to include other concn. ranges. The oxidised sample is mixed with a BaF_2 - graphite mixture (1 : 1) and pressed into an electrode (0·25 in. diam., 2 mm deep). Excitation of the sample is provided by high-current d.c. arc. Intensity ratios are calculated for the analytical line pairs and concn. determined from working curves.
NUCL. SCI. ABSTR.

4344. **Thermogravimetry of analytical pr tates. LXVI. Determination of hafnium.** Dautel and C. Duval (École Nat. Supérieu Chim., Paris, France). *Anal. Chim. Acta*, 195 (2), 154–159 (in French).—Various precipitant used for Hf^{4+} and the resulting compounds examined with the thermobalance to find the suitable means of gravimetric determination. following reagents yield ppt. that must be ig to HfO_2, and the minimum ignition temp indicated—aq. NH_3, 350°; air charged with 500°; tannin, 655°; *p*-hydroxyphenylarsonic 660°; cupferron, 670°. When pptd. as phosp HfP_2O_7 is obtained on heating the product to The basic selenite obtained by pptn. with S is of variable constitution until it is decompos HfO_2 at 680°, but if the ppt. is digested at 8 becomes $Hf(SeO_3)_2$ which is stable at 90° to The ppt. obtained with mandelic aci $Hf(C_6H_5{\cdot}CHOH{\cdot}CO_2)_4$, which is stable at 90° to and yields HfO_2 at 500°. The normal sel and the mandelate are the preferred compoun
H. N.

4345. **Extraction of thorium by a mixtu ethyl acetate and acetone.** M. Andjelković D. Rajković (Inst. for Technol. of Mineral Materials, Belgrade). Second U.N. Internat Conference on the Peaceful Uses of Atomic En Geneva, 1958, Rep. A/CONF.15/P/474, 10 The extraction is applicable to a wide range c concn. The method for the determination c is particularly efficient for materials with a Th content, using extraction with ethyl ace acetone as the separation method and Thoma colorimetric method for the final determina When the material contains larger amounts o the final determination can be carried out b gravimetric method. Besides Th, only U, Zr B were extracted among all the elements anal To avoid difficulties with Zr, the Zr is comp with tartaric acid and is not then extra Uranium is extracted quant., but during the c mination of Th with thoron it can be prese 25-fold excess without the error exceeding Bismuth does not interfere. The method is si sufficiently rapid, and applicable to routine From materials with a complex composition, m gram quantities of Th can be determined wit error usually $< \pm 5\%$. NUCL. SCI. ABS

4346. **Determination of thorium in low-grade** A. E. Suñer (Comisión Nacional de Energía Ató Buenos Aires). Second U.N. International ference on the Peaceful Uses of Atomic En Geneva, 1958, Rep. A/CONF.15/P/1581, 26 The analytical procedure used for separating i ferences has a separation factor (quotient o amount of interference before and after usin procedure) of 10^4. It consists in pptg. the Th HF, digesting with Na_2CO_3, extracting the Th diethyl ether, pptg. as iodate in an oxalic medium with a silver carrier ion, and spe photometric measurements with alizarin r (C.I. Mordant Red 3). The complete recove Th, in each of the steps of the analysis, has verified by using ^{234}Th, as well as by spectrop metry and gravimetry. The method was ap to the analysis of some minerals with satisfa results. NUCL. SCI. ABS

4347. **Quantitative analysis of thorium in erals and thoriferous products by a method bas radioactivity.** R. Collee, J. Govaerts and L. Wi (Univ. of Liége). Second U.N. Internat

ence on the Peaceful Uses of Atomic Energy, a, 1958, Rep. A/CONF.15/P/1701, 15 pp.— f résumé of the classical methods of quant. ination of Th in ores and thoriferous products n to show that a rapid, accurate and precise al method based on the radioactivity of Th be of great use. A method based on the use e characteristic spectrum of the thorium ation is presented. The preparation of the es and the instruments needed for the meas- nt are discussed. The experimental results eproducible and it is possible to detect Th nts of $<1\%$. NUCL. SCI. ABSTR.

8. **Determination of thorium in monazite ore eans of EDTA. I. Separation of thorium by itation with sodium iodate.** Saburo Kitahara hin Hara (Sci. Res. Inst., Tokyo). *Rep. Sci. Inst., Tokyo*, 1957, **33**, 340–342.—To the soln. ned by decomposing monazite ore with H_2SO_4 oxalic acid to transform Zr and Ti into com- s and then add $NaIO_3$ to precipitate Th. In procedure a small quantity of Th remains in oln. and considerable quantities of rare-earth nts are co-pptd. with thorium iodate.

Titration of thorium, with catechol violet as tor. Shin Hara and Saburo Kitahara. *Ibid.*, **33**, 343–349.—Decompose monazite ore with $_4$ and add $NaIO_3$ in the presence of 2% oxalic and 3 *N* HNO_3 until its concn. is $>8\%$, then This procedure precipitates almost all the s the iodate even if large quantities of SO_4^{2-} present. Dissolve the thorium iodate in dil. $_3$; decompose the oxalic acid with $KMnO_4$; mpose the $NaIO_3$ and the excess of $KMnO_4$ taneously with hydroxyammonium chloride. determine Th by titration with EDTA, with hol violet as indicator. Rare-earth elements, in large quantities, do not interfere. Zinc not interfere if large quantities of rare-earth ents are not also present. Avoid the inter- ce caused by Fe^{3+}, Al^{3+}, PO_4^{3-} and SO_4^{2-} by tg. the thorium iodate. CHEM. ABSTR.

49. **Sodium hypophosphate as a reagent for the ation of thorium, zirconium and niobium.** osta and L. Ravnik (J. Stefan Inst., Ljubljana, oslavia). Second U.N. International Con- ce on the Peaceful Uses of Atomic Energy, va, 1958, Rep. A/CONF.15/P/482, 11 pp.— ective procedure for the separation of Nb, Zr Th from most elements has been devised. It sed on the pptn. of these elements with sodium phosphate, with Ti as carrier. The pptn. from HCl is nearly quant. After eliminating other fering elements, Th is separated as the fluoride. nium is co-pptd. as barium zirconium fluoride a soln. in which Nb is complexed with HF. ium is finally isolated from the HF medium ne cupferrate. A detailed description of the edures for the determination of the isolated is given. A liquid - liquid extraction system mesityl oxide as solvent for separating Nb from HCl - HF medium is described, and data for the ibution coeff. are included. The advantage of system over others is that it requires a low n. of HF. NUCL. SCI. ABSTR.

50. **Rapid method for determining the concen- on of ammonium sulphate solutions.** J. R. taker and R. Hughes (Dept. of Food Technol., . of California, Davis, U.S.A.). *Nature*, 1959, 603–604.—From refractive index measure- ts of soln. of $(NH_4)_2SO_4$, the following equation, which avoids the necessity for density determinations, was developed—

$$Y = 0{\cdot}0403 + 41{\cdot}1(RI_1 - RI_2) + 309(RI_1 - RI_2)^2,$$

where Y is the molarity of the $(NH_4)_2SO_4$ soln., and RI_1 and RI_2 are the refractive indices of the soln. and of water, respectively. The temp. need not be known, and the over-all accuracy is $\pm 1{\cdot}0\%$ in the range 0·50 to 4·06 *M*. The sensitivity can be increased 10-fold by the use of an immersion refractometer, but the Abbé refractometer is adequate for soln. used for gradient elution and protein pptn. S. BAAR

4351. **Colour test for the detection of hyponitrites.** J. Vepřek-Šiška, V. Pliška and F. Šmirous (Dept. of Inorg. Chem., Tech. Univ., Prague, Czechoslovakia). *Analyst*, 1959, **84**, 119.—The test for hyponitrites described by Corbet (*Biochem. J.*, 1934, **28**, 1577) and by Rao and Rao (*Analyst*, 1938, **63**, 718) is investigated to determine its specificity and the optimum conditions for its application. It was found that the colour produced by reaction of hyponitrites with resorcinol and KIO_4 in acid soln. is not specific for hyponitrites but is for nitrites. The absorption spectrum of the red soln. has a characteristic maximum at 513 mμ, the position of which is independent of pH, but the extinction decreases with increasing pH. Other compounds of N that are oxidised by KIO_4 to nitrite therefore produce the same colour reaction with resorcinol and KIO_4 as does hyponitrite. As this, the only published reaction for hyponitrite, proves to be a reaction for nitrite, it is not possible to identify small amounts of hyponitrite formed in the presence of nitrite, oxyhyponitrite or hydroxylamine. A. O. JONES

4352. **Modality of the isotope dilution method of chemical analysis applied to the determination of phosphorus.** J. M. Arriaga e Cunha and J. Baptista (Estacão Agrónom. Nacional, Sacavém, Portugal). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/2332, 9 pp.—A constant amount of P is pptd. from two aliquots of an unknown labelled phosphate soln. to one of which a known wt. of inactive P is added. From the activities remaining in the supernatant soln., and from the wt. of P in the ppt., the P content of the soln. is calculated. The results obtained by the proposed method for the determination of P in a soln. with a known concn. of the element agreed closely with those obtained with the same soln. by the colorimetric method of Allen. The accuracy of the method must be checked when the determination of P by conventional methods is difficult. NUCL. SCI. ABSTR.

4353. **Potentiometric determination of phosphate ions.** Z. I. Ivanova and P. N. Kovalenko (Rostov-Don State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (1), 87–90.—Phosphate ions are determined by potentiometric titration with $Hg(NO_3)_2$ soln. with a mercury indicator electrode. With decrease of concn. of PO_4^{3-} the error increases, *e.g.*, from $+ 0{\cdot}7\%$ with 28·4 mg of PO_4^{3-} per ml to $- 20{\cdot}3\%$ with 2·6 mg per ml, with 0·161 *N* $Hg(NO_3)_2$; satisfactory results (error $\not> 2\%$) are obtained with $\not< 5$ mg of PO_4^{3-} per ml. The addition of ethanol decreases the error. The presence of colloidal substances (in soil extracts) does not influence the results. The errors in determining small concn. of PO_4^{3-} are much reduced by using the method of

double additions (*i.e.*, titrating, adding more phosphate soln. in two portions and titrating after each addition). The presence of 100-fold amounts of Cl^- does not cause interference. Phosphate and Cl^- can be determined successively. No interference is caused by the presence of Ca^{2+}, Mg^{2+}, Ba^{2+} or SO_4^{2-}. The pH of the soln. may be in the range 3 to 10. The method has been tested on minerals. C. D. KOPKIN

4354. **Paper chromatography of polyphosphates.** E. Heinerth (Henkel & Cie., Düsseldorf, Germany). *Z. anal. Chem.*, 1959, **166** (1), 37–39.—After separation of the phosphates by ascending chromatography with ethanol - water - trichloroacetic acid (80 ml : 20 ml : 5 g) and a little aq. NH_3, the bands, revealed by spraying with ammonium molybdate, drying, and spraying with a reducing soln., are cut out. The paper is destroyed by boiling with $HClO_4$, and the phosphates are hydrolysed by boiling the diluted soln. and determined spectrophotometrically. G. BURGER

4355. **Analytical chemistry of the fluorophosphates.** R. Klement and K. O. Knollmüller (Inst. f. anorg. Chem., Univ., München). *Z. anal. Chem.*, 1959, **166** (3), 193–201.—Phosphate, FPO_3^{2-} and $F_2PO_2^-$ may be detected in the presence of one another by paper electrophoresis in a buffer at pH 7. In the absence of any pptg. reagent, two spots are formed, one containing PO_4^{3-} and the other the fluorophosphate ions. If the initial soln. is spotted on a strip impregnated with uranyl acetate, PO_4^{3-} do not migrate. If the strip is impregnated with Pb acetate only $F_2PO_2^-$ migrate. Mixtures of PO_4^{3-} and fluorophosphates may be analysed by pptn. of Ag_3PO_4, filtration, removal of Ag^+ from the filtrate by ion exchange, boiling the eluate with HNO_3 and determining the PO_4^{3-} formed. T. R. ANDREW

4356. **Chemical microscopy of inorganic phosphorus - nitrogen compounds. Identification by their *o*-tolidine, ammonium and silver salts.** M. L. Nielsen and W. W. Nielsen (Res. and Engng Div., Monsanto Chemical Co., Dayton, Ohio, U.S.A.). *Microchem. J.*, 1959, **3** (1), 83–90.—Directions are given for the preparation of the *o*-tolidine, ammonium and silver salts of phosphoramidic, phosphorodiamidic (**I**), tri- and tetra-phosphenimidic acids. Eighteen photographs are given of the crystals, and it is shown that the tolidine salts serve for the identification of all the compounds except **I**, even in mixtures. Ammonium and silver salts are useful for the identification of **I**, and for the supplemental identification of the other compounds named. The effect of added substances such as decomposition products has not been studied, but it is noted that the absence of typical crystal forms must not be construed as indicating the absence of the species from the sample. R. E. ESSERY

4357. **Analytical applications of the reaction between sodium arsenite and ferricyanide catalysed by osmium tetroxide.** F. Solymosi (Inst. Inorg. and Anal. Chem., Univ., Szeged, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1958, **16** (3), 267–278 (in English).—*Cf. Magyar Kém. Foly.*, 1956, **62** (9), 318; *Anal. Abstr.*, 1957, **4**, 1214. W. J. BAKER

4358. **New (crystal phosphor) luminescence method of microchemical analysis. I. Detection of antimony.** K. P. Stolyarov and N. N. Grigor'ev (A. A. Zhdanov Leningrad State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (1), 71–74.—*The detection of Sb on ignited CaO.*—The method is based on formation of the crystal phosphor CaO.Sb igniting a bead of damp $CaCO_3$ in a platinu nichrome loop and adding a drop of test soln. igniting in a flame and inspecting in u.v. lig greenish-yellow luminescence is observed if present. The method will detect 1×10^{-6} Sb at a limiting dilution of 1 in 1×10^{10}. *detection of Sb with $K_4Fe(CN)_6$*—This is base the formation of the phosphor $K_4Fe(CN)_6.Sb$, is best carried out by drying a spot of reagent on a watch-glass, adding a drop of the test sol the hot dry spot, and allowing to dry. The cry formed luminesce with a yellow colour in u.v. l the detectable minimum is 0·001 μg of Sb limiting dilution of 1 in 1×10^7. Only As giv analogous reaction (detectable minimum, 0·1 limiting dilution 1 in $8{\cdot}3 \times 10^4$). Since elements form ppt. with $K_4Fe(CN)_6$, the Sb sh first be extracted from HCl soln. with diethyl e C. D. KOPK

4359. **Vanadametry. Determination of terv antimony: use of iodine chloride catalyst.** Bhaskara Rao (Dept. of Chem., Andhra U Waltair, India). *Z. anal. Chem.*, 1959, **165** 195–197 (in English).—Tervalent Sb is q oxidised with sodium vanadate in *N* H_2SO_4 i presence of ICl as catalyst. It is shown that reaction is completed in 5 min. at 98° to The excess of vanadate is then titrated with F soln. B. B. BAUMING

4360. **Spectrographic analysis of type-metal its antimony and tin content.** V. T. Ivanova E. M. Afanas'eva. *Sb. Trud. Vses. Nauch.-Is Inst. Goznaka*, 1957, (1), 236–242; *Ref. Z Khim.*, 1959, (1), Abstr. No. 1002.—Use the sa as a cylindrical rod ground to a flat surface. E the spectra with a carbon upper electrode i a.c. arc discharge at 4 to 5 amp. with an arc ga 2 mm. To prevent fusion of the test alloy, inc in the transformer circuit an interrupter so that time of arcing is 0·04 sec. with an interval o sec. Photograph the spectra in a medium spe graph with a slit width of 0·015 mm and an posure of 10 sec. Construct calibration cu from the lines (in A) Sb 2877·9 and Pb 28 Sn 2850·6 and Pb 2873·3. The determin concn. are Sb from 6 to 13% and Sn from 2 to Prepare standards by fusion of the pure metal 500°, with subsequent chemical analysis. choice of analytical lines and the conditions o discharge are studied. The analysis takes 30 C. D. KOPK

4361. **Determination of bismuth in lead and cable-sheathing alloys.** J. H. Thompson and B Peters (Materials Section, Test and Inspec Branch, P.O. Engng Dept., Fordrough L Birmingham, England). *Analyst*, 1959, **84**, 182.—The finely divided sample is dissolved in HNO_3 (1 : 2), the soln. is reduced to the lo practicable bulk and is then boiled with HCl ml); HBr is carefully added until the evolutio Br ceases, the cooled diluted liquid is filtered and washed $PbCl_2$ ppt. is discarded. The filtrat adjusted to 4 to 6 *N* with HCl and the resi Br is extracted with CCl_4. The aq. layer is extracted with a soln. of diethylammonium diet dithiocarbamate soln. in $CHCl_3$ (4 to 6×10 an oxidising mixture of H_2SO_4, HNO_3 and HCl added to the combined extracts, the liqui evaporated to fuming-point and finally heate copious fuming. To the cooled digest are ad

(1 to 2 ml), tartaric acid (5 g) and 20 ml of dil. (1:15), and, when the tartaric acid is ed, 20 ml of thiourea soln. [10% in dil. (1:15)]. After adjustment to a known vol. he thiourea soln. the extinction is measured mμ against water and referred to a calibra- raph. Results are good in the range 0·002 to A. O. JONES

. Organic reagents for salts of quadrivalent um. I. M. Korenman, V. G. Potemkina and Trusova. *Uch. Zap. Gor'kovsk. Univ.*, 1958, 09–111; *Ref. Zhur., Khim.*, 1959, (1), Abstr. 4.—A number of chromotropic acid azo dyes idied as reagents for V^{IV}. The most sensitive ons are found with dyes of the composition $)_2(OH)_2C_{10}H_3N:NR$, where R is $HO(Cl)C_6H_3$– $O_3SC_6H_4$– (**II**) or $H_2NC_6H_4$– (**III**). For the ion of V^{IV}, add 0·5 ml of 2 *N* aq. NH_3 to f test soln. containing 0·1 mg of V, centrifuge ppt. comprising accompanying cations with metavanadate, acidify the clear centrifugate cetic acid, add 10 to 15 mg of cryst. hydrazine te to reduce VO_3^- to VO^{2-}, 1 drop of a 0·1% ln. of reagent and 0·2 ml of 25% hexamine The limiting dilution is 1 in 4×10^6. Reagent the most specific. The detectable minimum using **I**, **II** or **III** is 0·25 μg of V per ml. of the dyes studied reacts with metavanadate. ossibility is shown of using these reagents for lorimetric determination of 20 to 100 μg of ml. C. D. KOPKIN

3. Coulometric titration with quinquevalent m. Determination of vanadium(V). S. L. s and D. M. Kern (Dartmouth Coll., Hanover, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (3), 98.—A procedure is given for the determina- f milligram amounts of V^V by titration with lytically generated U^V at pH 1 to 2·5 in an phere of N. The end-point is determined ometrically by a series of small increments of t. The apparatus is similar to that used for ration of Fe^{3+} with U^V (*cf.* Edwards and Kern, *Abstr.*, 1957, **4**, 1828). The error is $\simeq \pm 0{\cdot}3\%$; as low as 15 μg can be titrated. Titrations tures of Fe^{III} and V^V can be made at pH 1·5, ons being reduced simultaneously. W. J. BAKER

4. Decomposition of niobium-bearing ores and Aspects of the determination of niobium ocyanate. G. H. Faye (Dept. Mines Tech. ys, Ottawa). *Chem. Canada*, 1958, **10** (4), —The decomposition of Nb-bearing ores and by fusion with bisulphate and the subsequent metric determination of the Nb with thio- te gives low results in the presence of U. ttack with HF - HCl - H_3PO_4 is faster, gives te results in the presence of U, and offers a er of other advantages. To a 0·1 to 2-g e in a platinum dish add approx. 15 ml of HF, 10 ml of conc. HCl and 1 to 2 ml of H_3PO_4. rate the mixture to a paste. Cool and wash volumetric flask with *M* tartaric acid, filtering essary. Dilute with *M* tartaric acid and use ts for colour development by the method of and Marranzino (*cf. Anal. Abstr.*, 1955, **2**, CHEM. ABSTR.

5. Determination of sulphur in mixtures of n, potassium and magnesium chlorides. E. and T. W. Evett [U.K.A.E.A. (Research), C.37, Royal Arsenal, Woolwich, England]. .E. Analytical Method AERE-AM 5, 1959, 6 pp.—The method is based on that of Luke (*Anal. Chem.*, 1949, **21**, 1369). The S is converted into H_2S by a mixture of HI and H_3PO_2 in a special apparatus (illustrated) and then distilled into a soln. of Zn acetate. The S is then determined colorimetrically after reaction with HCl, *p*-phenylenediamine and $FeCl_3$ for 30 min. The extinction of the soln. is measured at 595 mμ and the content of S is derived from a standard curve. The error should not exceed ±5% and the method is suitable for the determination of 5 to 40 μg of S. It is essential to work in a sulphur-free atmosphere, as atmospheric contamination is difficult to avoid. G. J. HUNTER

4366. **Quantitative determination of traces of sulphides.** E. Hoffmann (Div. of Building Res., C.S.I.R.O., Melbourne, Australia). *Z. anal. Chem.*, 1959, **166** (3), 168–170 (in English).—The apparatus described comprises two small reaction vessels joined by a U-tube containing water. To each vessel, one containing the specimen and sodium azide soln., and the other containing only sodium azide soln., add iodine soln., and shake the apparatus gently. The vol. of N evolved in the tube containing the sample is proportional to the amount of S^{2-} present, and is measured as the difference in pressure in the two reaction vessels shown by the difference in the water levels of the two arms of the U-tube. This has been directly related to S^{2-} concn. for 0·05 to 0·6 μg of S^{2-}. A precision of ±25% is claimed. T. R. ANDREW

4367. **Determination of sulphur in pyrites concentrates, and the possibility of determining sulphur in volatile organic and inorganic substances.** Z. Řezáč and K. Straka (Forschungsinst. f. anorg. Chem. u. d. Verein f. chem. u. metallurg. Produktion, Ústí nad Labem, ČSR). *Z. anal. Chem.*, 1959, **166** (3), 161–168.—The finely ground sample (0·5 g) is mixed with 4 g of powdered manganese dioxide (containing 10% of KOH) in a porcelain crucible, then covered with a further 2 g of manganese dioxide and heated, first at ≃400° for 10 min. and then in a furnace for 15 min. at 800°. The cooled crucible and contents are boiled briefly with water, and the liquid is diluted to 250 ml and filtered through a dry paper into a dry beaker; the first fraction is rejected. An aliquot (50 ml) of the remainder is passed through a Wofatit P ion-exchange resin column (H^+ form) at a rate of 10 ml per min. The column is washed twice with water and the percolate and washings are titrated with 0·1 *N* NaOH to screened methyl red. Arsenic, P and Ca do not interfere, but about 95% of the S present as $PbSO_4$ and only about 20% of the S present as $BaSO_4$ is determined. On 32 specimens with a sulphur content of about 40% the standard deviation of the differences from the Senf and Schöberl method (*Angew. Chem.*, 1937, **50**, 338) was ±0·076. Encouraging results by this method are reported for sulphosalicylic acid, sulphanilic acid, thiourea and sulphathiazole. T. R. ANDREW

4368. **Rapid method of determining sulphur dioxide in hydrogen fluoride.** G. V. Rabovskiĭ, T. N. Egorova and O. P. Kasatkina. *Zavod. Lab.*, 1959, **25** (1), 36–38.—When a saturated soln. of $NaHCO_3$ containing iodine is used as an absorbent for determining SO_2 in gaseous HF the vol. of CO_2 liberated can be used to calculate the vol. of the gas which has been passed into the absorption vessel. The reaction of 1 mole of HF with $NaHCO_3$ yields 1 mole of CO_2, and the reaction of 1 mole of SO_2

with iodine and $NaHCO_3$ yields 4 moles of CO_2. The sample of gaseous HF is passed at a rate of 100 to 150 ml per min. into a vessel containing 100 ml of satd. $NaHCO_3$ soln. together with 1 to 5 ml of 0·01 *N* iodine and 3 to 5 ml of starch soln. until the absorbing soln. is decolorised. The vol. of CO_2 liberatde is measured by causing it to displace water from an aspirator bottle. The method is applicable to the determination of $<0{\cdot}01\%$ of SO_2 in gaseous HF with a relative error $>7\%$. G. S. SMITH

4369. **Determination of the sulphur dioxide content of flue gases from boilers.** B. A. Chertkov and D. L. Puklina. *Teploenergetika*, 1958, (9), 87–89; *Ref. Zhur., Khim.*, 1959, (3), Abstr. No. 8005.—The iodimetric method of determining SO_2 in flue gases is studied, the determination being carried out in a sample of the gas taken from the total gas flow. Good reproducible results are obtained and the accuracy is within 0·03 mg of SO_2 per litre. A study is also made of the factors influencing the magnitude of the error due to oxidation of the SO_2 to SO_3 by oxygen contained in the gas. To determine the average concn. of SO_2 in flue gas over an interval of time (3 to 40 min.), a method is developed consisting in drawing the sample of gas through a train of absorbers. The use of soln. of iodine or $KClO_3$ is recommended to take up the SO_2; in both cases very precise results are obtained. C. D. KOPKIN

4370. **Photometric determination of traces of selenium or tellurium in lead or copper.** C. L. Luke (Bell Telephone Lab., Inc., Murray Hill, N.J.). *Anal. Chem.*, 1959, **31** (4, Part I), 572–574.—Selenium and Te are isolated from large amounts of Pb or Cu by co-pptn. as the element with As. Selenium is then determined photometrically with 3:4:3′:4′-tetra-aminodiphenyl, and Te is determined as the diethyldithiocarbamate. The recovery of Se is quant., that of Te is $\simeq 93\%$.
P. D. PARR-RICHARD

4371. **Microgram determination of tellurium by an induced reaction with ferric phenanthroline.** C. O. Ingamells and E. B. Sandell (Univ. of Minnesota, Minneapolis, U.S.A.). *Microchem. J.*, 1959, **3** (1), 3–12.—The ferric complex of 1:10-phenanthroline (ferriin) is reduced to the ferrous complex (ferroin) by Te^{IV} in the presence of $K_2Cr_2O_7$, the red colour so produced being measured. *Ferroin soln.* (**I**)—Dissolve 0·05 g of 1:10-phenanthroline - ferrous perchlorate in 50 ml of 0·2% (v/v) H_2SO_4 soln. Allow to stand for several hours before use. The soln. is stable for 2 weeks. *Ferriin soln.* (**II**)—Shake 5 to 10 ml of **I** with 0·1 g of Mn-free PbO_2 till the soln. assumes a pure blue colour. Filter and re-pass the filtrate through the filter to ensure complete oxidation and removal of PbO_2. This soln. is unstable and is prepared immediately before use. *HNO_3 soln.* (1·0 M) (**III**)—Boil conc. HNO_3 for a few minutes to remove oxides of nitrogen. Dilute 20 ml to 300 ml with water and add 1 to 2 mg of urea. For the determination, evaporate the test soln. (containing <2 ml of conc. HNO_3 and >25 μg of Te^{IV}). Dissolve the dry residue in 1 ml of **III** with heating, and dilute to 20 ml with water. Add 1 ml of 1% NaCl soln. (except when Ag is present) followed by 1 ml of **II**, and mix. Add 2 ml of 0·015% $K_2Cr_2O_7$ soln., mix, make up to 25 ml and allow to stand for 10 min. Measure the absorption in a 1- or 2-cm cell at 475 mμ against a blank prepared at the same time with the same batch of **II**, and refer the result to a standard curve. Fixed acids are removed by evaporating the test soln. with excess of $NaNO_3$. Organic matter that is not pletely oxidised by HNO_3 is eliminated by f with KNO_3. Interference is strong from Cu^{II} and Ce^{III}, less strong with Au, Pd, Rh Sn, W and V. No interference occurs with concn. of Pb, Zn, Th, Hg, Bi, Fe, Mo, Hg, A Na, Ca and Se. The separation of trace amo of Te from interfering metals is briefly discu
R. E. ESSE

4372. **Rapid simultaneous potentiometric d mination of chromium, vanadium and iro carbides by means of titanium(III) chloride.** Dufek and H. Tůma (SVÚMT, Prague). *Listy*, 1959, **14** (3), 246–247.—The potentiom titration of Cr^{VI} to Cr^{III}, V^V to V^{III}, Fe^{III} to and Mo^{VI} to Mo^V can be carried out in a me containing $HClO_4$ and HCl. The first pote change corresponds to the reduction of Cr an (to V^{IV}), the second to the reduction of Fe, an third to that of Mo. After the addition of tar or citrate a further step corresponding to reduction of V^{IV} to V^{III} is observed. The am of standard soln. consumed in this titratio deducted from that used in the first potential ch to determine the amount of $TiCl_3$ correspondi the Cr. A derivative titration arrangement inum and S.C.E. electrodes connected wi galvanometer through a derivation adaptor sensitivity control) has been found advantag for the precise determination of potential cha *Procedure*—Dissolve 10 to 15 mg of the ca isolated from steel in $HClO_4$ (60%) (3 to 5 ml) several drops of conc. HNO_3 and evaporate. 30 min. dilute with several drops of H_2O, add HCl (15 ml) and titrate with $TiCl_3$ soln., stirring and bubbling with CO_2. After the pote changes corresponding to the reduction of Cr pl and Fe plus Mo, add Na citrate soln. (40%) (4 and titrate V^{IV} to V^{III}. J. ZÝ

4373. **Spectrochemical analysis of super- chromium for impurities.** C. G. Baird (De Standards Lab., Dept. of Supply, Maribyrn Victoria, Australia). *Appl. Spectroscopy*, 195 (2), 29–31.—A method for the determination of Pb, Fe, Al, Cu and Ag at concn. from 0·00 0·005% is described. The chromium is volati as chromyl chloride and the impurities are lected on a $CaSO_4$ matrix, which is then burned d.c. arc. Indium is added as an internal stan At an impurity level of 0·0005% the reproducib is better than $\pm 0{\cdot}0001\%$ for Mg, Pb, Fe, Cu Ag, and is $\pm 0{\cdot}0002\%$ for Al. K. A. PROCT

4374. **Colour reactions for chromates.** I. Korenman. *Uch. Zap. Gor'kovsk. Univ.*, (32), 145–147; *Ref. Zhur., Khim.*, 1959, (1), A No. 948.—The colour reactions of certain chr tropic acid azo dyes with $Cr_2O_7^{2-}$ in acid mediu studied. Many dyes are very sensitive reagent $Cr_2O_7^{2-}$, particularly in H_2SO_4, and may be use its detection. The most sensitive reaction is 2-(4-dimethylaminophenylazo)chromotropic with a limiting dilution of 1 in 4×10^5; no i ference is caused by the presence of Al, Zn, Hg, Bi, Cu, Ag, Co, Ni, Mn, or alkali or alka earth metals. Other oxidising agents (Mn Fe^{3+}) also cause a change in colour of the studied, but the sensitivity of these reactions is than with $Cr_2O_7^{2-}$. The ability of various oxi to react analogously to $Cr_2O_7^{2-}$ is a basis for sidering the change of colour of the dyes as due to their oxidation. C. D. KOPK

Photometric determination of molybdenum rrocyanide. L. T. Krasivskaya and R. P. sov. *Trudy Ural'sk. Lesotekhn. Inst.*, 1958, 3–38; *Ref. Zhur., Khim.*, 1959, (3), Abstr. No. -The intensity of the colour of colloidal soln. lybdenum^{VI} ferrocyanide in acid media Beer's law for concn. of Mo of 0·004 to 0·13 ml, but the soln. are stable for only 5 or 10 The colour intensity depends on the acid and is a max. in 0·15 *N* HCl. On this basis rimetric method for determining Mo is ped. The dissolution of the sample and the ion of Mo as the sulphide is carried out in the way. Make the soln. up to 100 ml, take a 0-ml aliquot so that the final concn. of Mo ≃0·03 mg per ml, dilute with water, add of HCl (1 : 1) and 1 ml of 0·1 *N* $K_4Fe(CN)_6$ and to 30 ml. Measure the extinction within 30 adding the ferrocyanide, with a blue filter. path length of 20 mm and a concn. of Mo mg per ml, the error is 0·002 to 0·004%. path length of 50 mm, ⩽0·002 mg of Mo may be determined. The described method nes as sensitive as the thiocyanate method.

C. D. Kopkin

Separation of molybdenum from vanadium traction as tributylammonium molybdate collate. M. Ziegler and H.-G. Horn (Inorg. Inst., Univ., Göttingen, Germany). *Z. anal.* 1959, **166** (5), 362–364.—After complexing and V with thioglycollic acid and tributyl- ium chloride, the molybdenum complex is ed with dichloromethane at pH 2, the omethane is evaporated and after wet on of the residue with 6 ml of conc. HNO_3 ml of HCl the Mo is pptd. with $Pb(NO_3)_2$ in esence of 50 ml of 50% ammonium acetate. $PbMoO_4$ is filtered off, washed with 2% O_3 soln. and H_2O and then dried at 500° to The average error is 0·4%.

B. B. Bauminger

Turbidimetric determination of traces of horus in tungsten and other metals. A. J. üs and M. Dvorszky (Forschungsinst. f. d. chtentech. Ind., Tungsram, Budapest, Hun- *Mikrochim. Acta*, 1959, (2), 202–209 (in n).—*Procedure*—The sample (1 g of WO_3) olved in 10 ml of H_2O containing 0·6 g of $_3$ by warming. The cooled soln. is neutralised HCl to phenolphthalein, and the following ts are added in the order indicated—10 ml of H_3 (10%), 2 g of NH_4Cl, 10 ml of Na_3AsO_4 containing 3 mg of As per ml) and 10 ml of soln. (10% in 5% NH_4Cl) dropwise with nt stirring. The mixture is diluted with H_2O ml and set aside for several hours. The ppt. red off on a G4 filter, washed with 5 ml of aq. 1%) and dissolved in 20% HCl (using a total f 20 ml). To the soln. are added 5 ml of acid (80%) and 5 ml of HBr (24%) and the re is evaporated by using an i.r. lamp. The tion in HCl and treatment with formic acid HBr are repeated. Finally, 0·5 ml of conc. is added to the residue and evaporated to y HBr; the residue is dissolved in 3·7 ml of (8 *N*) and washed into a 25-ml flask with a H_2O and the flask is immersed in a water t 20°. After 15 min., 2·5 ml of strychnine - dic acid soln. (specified) at 20° is added, the re is made up to vol. with H_2O, shaken, re- in the water bath for 1 hr. and the extinction red at 530 mμ; 5 × 10^{-5} % of P can be ined in 1 g. The determination of traces of P in copper, silver, magnesium, chromium, molybdenum, manganese, cobalt, nickel, aluminium, silicon and germanium is discussed.

B. B. Bauminger

4378. **Flame-photometric micro-determination of traces of sodium, potassium and calcium in tungsten metal and tungsten oxide.** A. J. Hegedüs, J. Neugebauer and M. Dvorszky (Forschungsinst. f. d. nachrichtentech. Ind., Tungsram, Budapest, Hungary). *Mikrochim. Acta*, 1959, (2), 282–293 (in German).—With the apparatus described and illustrated, W is volatilised by means of air saturated with CCl_4 at 450° to 600° and separated from alkali and alkaline-earth metals, which are then determined in the residue with a flame photometer. The completeness of the separation depends on the purity of the W or WO_3. The choice of boat in which the sample is volatilised is of importance, and it is shown that quartz boats are satisfactory, whereas platinum boats give low recoveries of Na. Gold, silver and molybdenum boats were unsatisfactory. The individual methods for the determination of traces of K, Na and Ca are discussed and the results tabulated. B. B. Bauminger

4379. **Dissolution of uranium metal and its alloys.** R. P. Larsen (Chem. Engng Div., Argonne Nat. Lab., Lemont, Ill.). *Anal. Chem.*, 1959, **31** (4, Part I), 545–549.—The general dissolution characteristics of U are described and methods for dissolving the metal and its alloys are reviewed. The following solvents are considered—HNO_3, HCl, aqua regia, $HClO_4$, H_2SO_4, HF, H_3PO_4, heavy-metal salts, ethyl acetate with Br or HCl and NaOH - H_2O_2. A table of common uranium alloys and their appropriate solvents is given; the choice of solvent depends to some extent on the subsequent determinations to be performed on the soln.

P. D. Parr-Richard

4380. **New spectrophotometric method for the determination of uranium.** F. da Silva and L. de Moura (Comissão de Estudos da Energia Nuclear, Lisbon). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/1814, 17 pp.—Small amounts of U_3O_8 are determined by making use of the displacement of the lactone form of Rhodamine B (C.I. Basic Violet 10) to the quinone form by the action of uranyl salts of carboxylic acids sol. in benzene. The colour is determined spectrophotometrically; Beer's law is obeyed for 5 to 80 μg of U_3O_8 per ml. The best operating conditions are selected and the main sources of interference considered; these are Fe, Sn, Mn, Sb and Tl. Since Cu and Al do not interfere, the classical scheme of separation may be followed. Some results for minerals poor in uranium are shown in comparison with those obtained by other methods.

Nucl. Sci. Abstr.

4381. **Isotopic analysis of uranium by emission spectroscopy.** G. V. Wheeler (Phillips Petroleum Co., Atomic Energy Div., Idaho Falls). Conf.: "Modern Approaches to the Isotopic Analysis of Uranium." Chicago, Feb. 5–7, 1957. Rep. TID-7531(Pt.2)(Del.), 25–31.—The isotopic analysis of U by emission spectroscopy has been investigated at the Idaho Chemical Processing Plant in three phases; a literature search, an investigation with the equipment available at the Idaho Chemical Processing Plant, and an investigation with high-resolution direct-reading equipment available commercially. These investigations are discussed and the conclusion is drawn that though more work

remains to be done, U can be assayed for ^{233}U, ^{235}U, ^{236}U and ^{238}U by emission spectroscopy with a precision approaching that of mass spectrometers but at much lower cost. NUCL. SCI. ABSTR.

4382. **Isotopic analysis of uranium by the emission spectrograph.** R. E. Kupel and W. Tichy (General Electric Co., Aircraft Nuclear Propulsion Dept., Cincinnati, Ohio). Conf.: "Modern Approaches to the Isotopic Analysis of Uranium." Chicago, Feb. 5–7, 1957. Rep. TID-7531(Pt.2)(Del.), 17–24.—The method is suitable for the analysis of U in the 90% enrichment range. Two graphite electrodes are loaded with an equal mixture of graphite and U and excited in an a.c. arc. The resulting spectra are monitored on a densitometer to obtain the relative intensities of the 4244·37-A line and the 4261·51-A line. The ratio of the relative intensities of these two lines is then applied to a concn. curve and the percentage of ^{238}U is obtained from the curve. A known amount of material is weighed and dissolved in HNO_3. A measured volume of soln. is evaporated on a stainless-steel disc and the α-activity is obtained with a proportional counter. The ^{234}U (%) is calculated from the α-activity, the ^{238}U (%) is obtained as described above, and the ^{235}U (%) is then obtained by difference, assuming that only the three isotopes are present. The accuracy of this method compares favourably with the mass-spectrometer method. The precision of the method at the 95% confidence level is ±0·30%. NUCL. SCI. ABSTR.

4383. **Uranium-235 abundance by γ-spectrometry.** S. A. Reynolds and J. S. Eldridge (Oak Ridge National Lab., Tenn.). Conf.: "Modern Approaches to the Isotopic Analysis of Uranium." Chicago, Feb. 5–7, 1957. Rep. TID-7531(Pt.2)(Del.), 47–52.—Techniques are described for determining ^{235}U abundance by measurement of the intensity of 0·18-MeV γ-radiation. One method involves the measurement of the ratio of the intensity of 0·18-MeV radiation to that of ≃0·1-MeV radiation. The preferred technique consists in the chemical separation of U, followed by direct counting of the 0·18-MeV photons. Application has been made in analysis of U samples of abundances in the range of 0·05% to 93%. The accuracy appears to be better than 3% if the abundance is between 0·7% and 100%. Data in the self-absorption of 0·18-MeV radiation in uranium oxide are presented. NUCL. SCI. ABSTR.

4384. **A γ-counting method for the determination of uranium-235 in enriched uranium.** U. L. Upson and D. G. Miller (General Electric Co., Hanford Atomic Products Operation, Richland, Wash.). Conf.: "Modern Approaches to the Isotopic Analysis of Uranium." Chicago, Feb. 5–7, 1957. Rep. TID-7531(Pt.2)(Del.), 32–46.—The ^{235}U can be detected and measured in the presence of other U isotopes and their daughters by selectively measuring its 184-keV γ-radiation. The determination of the ^{235}U content of highly enriched uranium in aluminium alloys is described. In this case it is possible to do the counting on small vol. of dissolved samples of the alloy without separation of the aluminium or of the uranium daughters. A precision of ± 1·2%, at the 95% confidence level, is possible by the technique described. The effects of daughter growth and of separation of the U from the Al are discussed, as well as other methods for improving the precision. NUCL. SCI. ABSTR.

4385. **Radiochemical method for the determination of uranium in rocks by means of fractional extraction of thorium-234 with hexone [*isobutyl m* ketone].** G. Alberti, C. Bettinali, F. Salvett S. Santoli (Chem. Lab., Geomineral. Div., C.N. Inst. of Gen. and Inorg. Chem., Univ. of R *Ann. Chim., Roma*, 1959, **49** (1), 199–204.—U ium-238 is in equilibrium with its direct radioa decay product, ^{234}Th, which in turn establish equilibrium with ^{234}Pa within ≃5 min. To a tain the uranium content of rocks, the ^{234} separated from interfering radio-elements by p ential extraction with *iso*butyl methyl ketone the β-emission of the ^{234}Pa is counted. The r are then compared with those obtained by sim treating standard soln. of uranyl nitrate. method is rapid and is suitable for concn. of low as 0·005%. No interference is shown b topes of Pb and Bi, or by PO_4^{3-}, SO_4^{2-} and Cl^- A. G. Coo

4386. **Direct measurement of uranium thorium by γ-spectroscopy.** C. Brooke, E. Picc and G. Poulaert (Univ. Libre, Brussels). *Soc. Belge Géol. Paléontol. Hydrol.*, 1958, **6** 315–328 (in French).—The development of spe metric methods for determining U and Th an usefulness of the techniques in geochemical geochronological research are discussed. Ex ments are described in which Th and U are d mined simultaneously in weighed artificial mix by γ-spectroscopy. The error increased whe concn. decreased and also increased when the Th ratio differed from 1. The error ranged 1% for concn. near 0·1 g per g to 10% for con several mg per g. To maintain the error in Th U determination at less than 10%, the U t ratio should be between 0·1 and 10. For s densities of about 0·2 g per cu. cm the determin is not affected by variations in the density or n of the sample. The determination can be ma 30 min. directly on a powder of the mineral or NUCL. SCI. ABS

4387. **Analysis of uranium, thorium and conium.** G. Almássy, M. Ördögh and A. Sc (Central Res. Inst. for Physics, Budapest). S U.N. International Conference on the Pea Uses of Atomic Energy, Geneva, 1958, A/CONF.15/P/1718, 18 pp.—In investigatin physico-chemical conditions for the tributyl phate extraction of U, the optimum conc NO_3^- in the aq. phase in which the separation by single extraction amounted virtually to 100% determined. The extraction was carried o tributyl phosphate - kerosine and the U re-extr in dil. ammonium carbonate soln. The U co was determined by the reduction of U^{VI} to and subsequent oxidising with Fe^{III}, which c measured by the light absorption of the dipyridyl complex equiv. to U at 500 mμ. Fo chromatography of Th it has been found that the solvents indicated in the literature, th produces an elongated spot on a cellulose co or on paper. This spot becomes more compa increasing the acid and water content of the so The optimum composition of the solvent determined: 8 *N* HNO_3 was shaken with an amount of diethyl ether and after separation two phases the ether phase was used. Amon colorimetric determinations of Th, the t method was improved for the analysis of or reducing Fe^{III} with ascorbic acid. The sensitiv the method is increased by ≃ 70% by the ad of methanol. Zirconium and hafnium ca separated selectively by mandelic acid. The containing 0·2 to 3 mg of Zr, is dissolved in

c. H_2SO_4 and the mandelic acid, bound quant. e Zr can be oxidised to CO_2 and water with $_2O_7$ soln. In the presence of 2 to 20 mg of ie oxidation can be carried out in excess of O_4 soln. in a fairly alkaline medium. The yellow zirconium - morin complex, which a green fluorescence in u.v. light, is suitable e photometric determination of Zr. Measure- s can be carried out between 420 and 470 mμ.

Nucl. Sci. Abstr.

8. **Use of radioactive isotopes in studying the l distribution of elements in direct current arc a during spectrochemical determination of ities in uranium.** É. E. Vaĭnshteĭn and Yu. I. ev (V. I. Vernadsky Inst. of Geochem. and Chem., Acad. of Sci., Moscow). *Intern. J. Radiation and Isotopes*, 1959, **4** (3–4), 179– -Uranium oxide with various added com- is labelled with γ-active nuclides was examined rographically. In addition to normal photo- ing of the discharge, the distribution of the l element was determined by observation of -activity with a pin-hole camera. The distri- ns of Sb, Ga and Cu in various proportions studied particularly from the aspect of the use $_2O_3$ as a carrier in the determination of Sb; istributions of Ag, and of Na, Ca and Ga were examined. It was concluded that, (*i*) the bution of an element depends upon its vola- (*ii*) the distribution of a micro-constituent vs that of a macro-constituent when both ituents are being removed from a base of low ility, and (*iii*) the materials used as carriers in elements that are symmetrically distributed arc between the electrodes.

G. J. Hunter

9. **Separation of traces of rare-earth elements in um by ion exchange.** H. A. Braier (Comisión onal de Energía Atómica, Buenos Aires). d U.N. International Conference on the Peace- Uses of Atomic Energy, Geneva, 1958, Rep. ONF.15/P/1568 (Rev.1), 8 pp.— A soln. of +, with traces of Eu, in a H_2SO_4 medium is d through a bed of cationic resin, so that the ic complex of the UO_2^{2+} passes through and the earth elements are retained. The remainder e U is eluted with H_2SO_4, and the rare-earth ents are eluted with HCl. The final determina- is made spectrographically. The fixation of y the resin and its subsequent elution is ob- d by adding ^{154}Eu, which acts as a tracer. method is promising for the determination e rare-earth elements in uranium because it is ler than those described in the literature.

Nucl. Sci. Abstr.

0. **Determination of isotopic ratios in uranium fluoride using dual ionisation chambers.** J. H. ns, E. C. Denny and W. G. Schwab (Oak Ridge ous Diffusion Plant, Tenn.). Conf.: "Modern oaches to the Isotopic Analysis of Uranium." ago, February 5–7, 1957. Rep. TID-7531-)(Del.), 7–16.—A dual ionisation chamber od was investigated for possible use in deter- ng small differences in ^{235}U concn. in uranium fluoride. Two ionisation chambers are con- d in ion polarity opposition, and the differential current is collected on a capacitor. Instru- tation automatically converts the information igital form and records the time required to ct a fixed charge. Also studied was an altern- method of measuring the ratios of times red for the voltage to change by a fixed interval.

Nucl. Sci. Abstr.

4391. **A new electrolytic carrier for the determination of uranyl ion.** C. J. Sambucetti and A. Gori (Comisión Nacional de Energía Atómica, Buenos Aires). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/1576, 25 pp.—The polarographic behaviour of the complex formed between UO_2^{2+} and tiron (disodium catechol-3:5-disulphonate) is reviewed, and the best conditions for the determination of the U present in this medium are established. The electrode reaction is reversible in the pH range from 2 to 3·5. The $E_{\frac{1}{2}}$ varies with the pH; its values range from 0·222 to 0·243 V (*vs.* the S.C.E.), with a tiron concn. of 0·05 *M*. The possible electrode reaction is indicated.

Nucl. Sci. Abstr.

4392. **Spectrophotometric determination of contamination in nuclearly pure uranyl nitrate.** J. Nowicka-Jankowska, A. Gołkowska, I. Pietrzak and W. Zmijewska (Polish Acad. Sci., Inst. Nucl. Res., Warsaw). Inst. of Nucl. Res., Warsaw, Rep. No. 45/VIII, 1958, 42 pp.—Methods of determining trace amounts of Ni, Al, Si, Mo, Cr and Fe in uranyl nitrate are described. Nickel is determined with dimethylglyoxime, Al with haematoxylin and Si as molybdenum blue. Molybdenum may be determined by the thiocyanate method, or with dithiol, Cr is determined with diphenylcarbazide, and Fe with 2:2′-dipyridyl. Nucl. Sci. Abstr.

4393. **Improved method for the determination of trace quantities of plutonium in water.** J. Kooi, U. Hollstein and M. Schlechter (Reactor Centrum Nederland, The Hague). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/550, 18 pp.—The method is based on the extraction of the cupferron complex following a concentration step. It was found that bismuth phosphate may advantageously be used as the co-pptg. agent. Measurements of the extraction, with $CHCl_3$, of Bi, Fe and Pu as cupferrates were performed. By properly adjusting the pH, a good separation of Bi and Pu may be achieved. A substantial reduction in the amount of Fe was found to be possible, reducing sample-size and self-absorption losses in α-counting. The yield of Pu compares favourably with that obtained by other methods.

Nucl. Sci. Abstr.

4394. **Analysis for neptunium-237 in nitric acid solutions.** B. B. Murray (E. I. du Pont de Nemours & Co., Savannah River Lab., Augusta, Ga.). *U.S. Atomic Energy Comm.*, Rep. DP-316, 1958, 14 pp.—A method was developed for the determination of ^{237}Np in HNO_3 soln. that contained U, Pu and fission products. Neptunium was extracted with 2-thenoyltrifluoroacetone after reduction to the quadrivalent state with ferrous sulphamate. The α-activity of the separated Np was then measured with an α pulse height analyser, and the degree of separation of Np from other α-emitters was determined. The precision of the analysis depended on the total amount of ^{237}Np present in the sample. A coeff. of variation of 2 to 5% was observed for samples that contained more than 180 α-disintegrations of Np per min. For activities in the range of 10 disintegrations per min., the coeff. of variation was from 10 to 25%. Nucl. Sci. Abstr.

4395. **Microcrystalloscopic reactions for halogens using organomercury compounds.** I. M. Korenman and A. A. Belyakov. *Uch. Zap. Gor'kovsk. Univ.*, 1958, (32), 85–91; *Ref. Zhur., Khim.*, 1959, (1),

Abstr. No. 975.—The microcrystalloscopic reactions of Cl^-, Br^- and I^- with 23 mono- and di-mercurated compounds of aniline and its derivatives, toluidines, quinoline and 2-aminopyridine are studied. Several of these compounds show sensitive and specific reactions with the halides, the most sensitive being the mono-mercury compounds. The sensitivity increases from Cl^- to I^-, while the max. crystal size decreases. Many of the crystalline ppt. show intense pleochroism. C. D. KOPKIN

4396. **Emission spectrographic analysis of fluorine with reference to the calcium fluoride band.** V. R. Ognev. *Trudy Sibirsk. Fiz.-Tekh. Inst., Tomsk. Univ.*, 1958, (36), 281–286; *Ref. Zhur., Khim.*, 1959, (9), Abstr. No. 31,048.—Fluorine is determined in bone and in materials the basic components of which are SiO_2 or NaCl. *Procedure*—Mix the sample with CaO (1:1) and excite the spectra in an a.c. arc between two carbon electrodes arranged at an angle of 90° in a horizontal plane. Calibration curves are constructed with the bands CaF (5291 A) and CaO (5473 A). The probable error is ± 6%. In the presence of large amounts of Ca the sample need not be mixed with CaO. K. R. C.

4397. **Determination of fluorine in beryllium compounds.** J. L. Huguet (Comisión Nacional de Energía Atómica, Buenos Aires). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/1582, 10 pp.—The applicability of titrimetric methods to the micro- and macro-determination of F in beryllium compounds was studied. A study is presented of the effect of various factors on the separation of F by distillation, and a technique is designed which affords means of obtaining distillates having characteristics compatible with the application of the titrimetric methods. NUCL. SCI. ABSTR.

4398. **Rapid determination of fluorine, sulphur, chlorine and bromine in catalysts with an induction furnace.** A. L. Conrad, J. K. Evans and V. F. Gaylor [Chem. and Phys. Res. Div., The Standard Oil Co. (Ohio), 4440 Warrensville Center Rd., Cleveland, Ohio]. *Anal. Chem.*, 1959, **31** (3), 422–425.—Rapid combustion methods are described for determination of F, S and Cl, and S and Br. Fluorine is volatilised as fluorosilicic acid in the presence of silica gel and an accelerator (iron) and absorbed in dil. NaOH soln. (pH 9 to 9·5); F^- are then titrated with $Th(NO_3)_4$ soln. at pH ≃3, with Na alizarinsulphonate as indicator; the end-point is found spectrophotometrically at 520 mμ. To determine S and Cl, the Cl is absorbed in aq. 0·005 *N* $AgNO_3$ at pH 1 and the excess of Ag is titrated amperometrically; S, which is not retained by 0·005 *N* $AgNO_3$, is titrated continuously, as it is evolved, by KIO_3 soln. For the determination of S and Br, the Br is absorbed in cold CCl_4, extracted with ice-water and titrated with $Na_2S_2O_3$ soln. after addition of KI and H_2SO_4; S is determined as before. P. D. PARR-RICHARD

4399. **Micro-method for determining fluorine monoxide.** E. A. Peregud and B. S. Boĭkina (State Scientific-Res. Inst. of Work Hygiene and Occupational Diseases, Leningrad). *Zhur. Anal. Khim.*, 1959, **14** (1), 141–142.—Microgram amounts of F_2O in air are determined by drawing 1 litre of the air in 20 min. through an indicator tube containing silica gel moistened with a soln. of fluorescein containing KBr and K_2CO_3 (*cf. Zhur. Anal. Khim.*, 1957, **12**, 513); F_2O liberates Br from the KBr, with subsequent formation of the red tetrabro fluorescein. The length of the coloured zon proportional to the concn. of F_2O. C. D. KOPKI

4400. **Determination of chloride in urani bearing materials by a potentiometric titra method.** P. G. Laux and E. A. Brown (National L Co. of Ohio, Cincinnati). *U.S. Atomic Energy Com* Rep. NLCO-751, 1958, 39 pp.—The metho suitable for the determination of Cl^- in the rang 0·005 to 0·15% in uranium-bearing feed materi The titration is carried out to a predetermi equivalence potential in a methanol - HNO_3 (1 medium. A silver wire - saturated calomel e trode system is used. The method is rapid si no preliminary separation of the Cl^- is required. major interferences are caused by Fe^{III}, MnO_4^-, NO_2^-, Br^- and I^-. The interference all ions except Br^- and I^- is prevented by addition of ascorbic acid or aq. NH_3. A c parative study of the potentiometric and turb metric methods was made. Uranium ore conc trates, HNO_3 and aq. feed samples were used in study. Satisfactory agreement was obtained tween the two methods. NUCL. SCI. ABSTR

4401. **Iodimetric assays. VI. The determinat of iodide ions in the presence of bromide and chlor** L. Szekeres and E. Kardos. *Magyar Kém. La* 1958, **13** (10–12), 447.—Iodide is oxidised with C BrO^- and the excess of oxidant is selectiv reduced with H_2O_2, Na formate, Na oxalate, urea ethanol; IO_3^- are then determined iodimetrica *Procedure*—To a sample of 0·01 to 0·025 *N* KI (5 10 ml) add approx. 0·1 *N* KBr - Br (10 to 25 and $NaHCO_3$ (3 to 5 g) and dilute with a few m H_2O. After 1 to 2 min. add ethanol (5 to 15 and heat for 15 min. on a water bath. Dilute soln. to 100 ml, acidify with 10 *N* HCl (10 ml) a titrate the iodine with 0·02 to 0·1 *N* Na_2S_2 The presence of Br^- or Cl^- has no effect on result. The error is < 0·7%. G. SZAB

4402. **X-ray fluorescence method for determin iodide in photographic processing solutions. Iod concentration technique.** D. Owerbach, R. Hoefler and H. J. Klem (Colour Technol. D Eastman Kodak Co., Rochester, N.Y., U.S. *Anal. Chem.*, 1959, **31** (4, Part 1), 579–581.— this method concn. of iodide as low as 1 mg litre can be determined with a coeff. of variatio ≃ ± 4%. The iodide is pptd. with a slight ex of $AgNO_3$, filtered off and dried, and the ppt analysed against a known standard. The eff of varying concn. of bromide at different iod levels have been studied and some possible in ferences in the X-ray technique are discussed. K. A. PROCTO

4403. **New reagents for the detection of te netium.** F. Jasim, R. J. Magee and C. L. Wil (Dept. of Anal. Chem., Queen's Univ., Belfa *Talanta*, 1959, **2** (1), 93–95.—Four colorime reagents for technetium are described. *Proced with potassium ethylxanthate*—To 10 mμl of the t soln. add 50 mμl of satd. aq. potassium ethylx thate soln. and 50 mμl of 2 *N* HCl. The prese of Tc^{IV} or Tc^{VII} is indicated by the immedi formation of a purple colour, which is reac extractable into CCl_4. The limit of identificat is 0·02 μg of Tc; Mo interferes. *Dimethylglyox and $SnCl_2$*—To 100 mμl of the test soln. add mμl of satd. dimethylglyoxime soln. in 9

ol and 50 mμl of $SnCl_2$ soln. (15 to 30% in HCl). A green colour indicates the presence IV or Tc^{VII}. The limit of identification is g. *Thiourea*—To 100 mμl of the test soln. 0 mμl of 2 *N* HNO_3 and 50 mμl of 10% aq. rea soln. An orange - red colour confirms the ıce of Tc. The limit of detection is 0·04 μg. *N in HCl*—To 100 mμl of the test soln. add ıl of 6 *N* HCl and 40 mμl of aq. KSCN soln. . Heat in boiling water for 5 min. A purple confirms the presence of Tc^{IV}.

W. T. Carter

4. **Separation of technetium from rhenium by electrophoresis.** R. A. Guedes de Carvalho ssão de Estudos da Energia Nuclear, Oporto, gal). Second U.N. International Conference e Peaceful Uses of Atomic Energy, Geneva, Rep. A/CONF.15/P/1810, 5 pp.—The separa- is achieved by reduction of Tc^{VII} to Tc^{IV}, is cationic or does not migrate, while Re^{VII} affected by the reductant and maintains its ic character. Among several reductants d, buffered soln. of hydrazine hydrate and ate (pH = 8 to 9) and hydriodic acid permitted ood and rapid separations of the two elements.

Nucl. Sci. Abstr.

5. **Separation of iron from certain metals by xchange chromatography.** R. N. Golovatyĭ. *Khim. Zhur.*, 1958, **24** (3), 379–383; *Ref. Khim.*, 1959, (1), Abstr. No. 896.—The bed method for separating Fe^{3+} from Ca, Mg, In, Co, Ni, Cu, Ti, Zn and Al is based on the tion of a stable anionic complex of Fe^{3+} is not adsorbed by a cationite in the H form. EDTA (disodium salt) (**I**) as the complexing nt from a burette, determining the moment of letion of the complexing of Fe by the change our of the soln. containing KSCN or sulpho- lic acid. To separate Fe from Al, Mg, Ba, u and Ni, acidify the soln. to 0·01 *N* with HCl, or 5 drops of 10% KSCN soln., and then **I** till the thiocyanate complex is decolorised. the resulting soln. through a column of diam. containing 10 g of Wofatit (H form) at a of 3 to 4 ml per min. All the cations except are adsorbed by the resin. Elute the adsorbed vith 3 *N* HCl. The described method can be or the separation of Fe from Co if the pH of the is first adjusted to ≯2. Elute Co with 3 *N* nd determine it by the method described earlier l, *"Kompleksonŭ v Khimicheskom Analize"*, nostr. Lit., 1955, pp. 86, 94]. To separate and Cr^{3+} from Ti^{4+}, add **I** to the soln. until Fe r are bound in a stable complex not destroyed tionites at pH 1 to 10, heat the soln. to between nd 60°, cool, and pass the soln. through a n of cationite. Elute the adsorbed Ti with 2 *N* H_2SO_4 containing 1% of oxalic acid, e the oxalic acid with permanganate, and nine Ti photometrically.

C. D. Kopkin

6. **Determination of various forms of iron in** Yu. P. Trusov (V. I. Vernadskiĭ Inst. of hem. and Anal. Chem., Acad. of Sci., USSR, ow). *Zhur. Anal. Khim.*, 1959, **14** (1), 139–140. rous, ferric and pyritic Fe are determined in gle sample by heating the coarsely ground e (0·5 or 1 g) in a stream of CO_2 with a mixture ml of H_2SO_4 (1:2) and 10 ml of HF, cooling, ng the mixture into about 80 ml of satd. $_3$ soln., and titrating with 0·1 *N* $KMnO_4$ to nine Fe^{2+}; Fe^{2+} plus Fe^{3+} is determined by ng off the pyrites (which is not attacked by this treatment), washing it with water, adding to the filtrate 15 ml of 25% NH_4SCN soln. and titrating the Fe with 0·1 *N* $Hg_2(NO_3)_2$; Fe^{3+} are found by difference. The residue of pyrites is ignited at about 800°, the Fe_2O_3 is fused with $K_2S_2O_7$, the melt dissolved by heating with H_2SO_4 (1:20), and Fe^{3+} are titrated with $Hg_2(NO_3)_2$. The method may be used for rocks not containing considerable amounts of oxidising - reducing substances.

C. D. Kopkin

4407. **Quantitative spectrographic analysis of ferrotitanium.** A. A. Tsimbal and V. Lichko. *Sudostroenie*, 1958, (7), 67–68; *Ref. Zhur., Khim.*, 1959, (2), Abstr. No. 4362.—Grind an average sample (20 g) and sieve. Mix 1 g of sample and 1 g of copper powder and press into a briquette with a pressure of 4000 kg per sq. cm, and clamp the briquette opposite a carbon electrode. Excite the spectra in a high-voltage spark discharge (circuit capacity 0·01 μF, self-induction zero, discharge gap 3 mm, and working gap 2 mm), and photograph in a medium spectrograph with an exposure of 100 sec. and a preliminary ignition of 30 sec. Construct calibration curves from the lines (in A)—Ti 3653·4 and Fe 3631·4, Si 2881·5 and Fe 2872·3, and Al 2816·1 and Fe 2831·5. To determine Cu, use a briquette prepared from 1 g of sample and 1 g of Al, and analytical lines Cu 3273·9 A and Ti 3653·4 A. The analytical error is ≯ 2·0%.

C. D. Kopkin

4408. **Electrolytic isolation of non-ferrous inclusions in steel with the use of a modified Klinger and Koch apparatus.** L. Brháček, J. Janáček and A. Šmrhová (Res. Inst. VŽKG, Ostrava, Czechoslovakia). *Hutn. Listy*, 1959, **14** (1), 54–55.—A modification of the Klinger and Koch apparatus (*Arch. Eisenhüttenw.*, 1938, **11**, 569) permits shortening of the time required for the isolation of inclusions by means of the high current density of 0·10 amp. per sq. cm. A detailed description of the apparatus and procedure is given. For samples with surface areas of 30 to 50 sq. cm, a current of 1 to 2 amp. and an electrolysis time of 24 hr. are recommended. A suitable anolyte is Na citrate (40 g), citric acid (10·5 g), KBr (15 g), NaCl (20 g) and KI (1 g) in 1 litre of H_2O, and a suitable catholyte is citric acid (86 g) and *N* Na_2CO_3 (1 ml) in 1 litre of H_2O; 500 ml of electrolyte is recommended per g of dissolved sample.

J. Zýka

4409. **Determination of copper in steel by amperometric titration with sodium diethyldithiocarbamate.** Yu. I. Usatenko and F. M. Tulyupa (F. É. Dzerzhinskiĭ Dniepropetrovsk Chem. Tech. Inst.). *Zavod. Lab.*, 1959, **25** (3), 280–283.—The sample (1 g) is heated with 20 to 30 ml of dil. HCl (1:1), and the soln. boiled with 2 to 3 ml of conc. HNO_3 (5 ml if W is present); 25 to 30 ml of water is added, and the soln. is boiled to remove oxides of N, and Cl, then neutralised with aq. NH_3 and HNO_3, cooled, and diluted to 100 ml. An aliquot (10 ml) is mixed with 2 ml of 0·1 *M* $Pb(NO_3)_2$ (to serve as an indicator), 0·5 g of K tartrate (neutral or acid salt) and 1 g of Na acetate. If a white cloudiness (Pb tartrate) does not appear a further amount of Na acetate is added. A few drops of satd. NH_4Cl soln. are added to dissolve the ppt. and the soln., after being diluted to 50 ml, is titrated amperometrically with 0·2% Na diethyldithiocarbamate soln. at an applied potential of +0·8 V *vs.* the S.C.E. at a rotating platinum electrode. When W is present the WO_3 is filtered off and washed with

very dil. HCl soln. before the neutralisation with aq. NH_3. The tartrate present prevents interference by Cr and Mn. Large amounts of Ni, Mo and V do not interfere. G. S. SMITH

4410. **Determination of copper in certain high-alloy steels.** C. R. Elliott, P. F. Preston and J. H. Thompson (Materials Sect., Test & Inspection Branch, P.O. Engng Dept., Fordrough Lane, Birmingham, England). *Analyst*, 1959, **84**, 237–239.—The sample is dissolved in dil. H_2SO_4 (or with stainless steel in dil. H_2SO_4 with subsequent oxidation of residual carbides with HNO_3), the liquid is heated to fuming, diluted to a known vol. and an aliquot, suitably diluted, is extracted with 0·5% dithizone soln. and finally with CCl_4 to remove residual dithizone. The combined extracts are heated with a mixture of H_2SO_4, HNO_3 and $HClO_4$ to oxidise organic matter. Citric acid soln. is added, the pH is adjusted to 8 to 9·5 with aq. NH_3, and the liquid is treated with 0·5% bis*cyclo*hexanone oxalyldihydrazone soln. in 50% ethanol (5 ml) at 20°. After development of the colour the liquid is diluted to vol., and its extinction is measured at 595 mμ against water, and referred to a calibration graph prepared from a standard soln. (1 mg of Cu per 10 ml). The most useful range of Cu in the final soln. is $\simeq$ 40 to 200 μg per 100 ml. The precision is satisfactory. A. O. JONES

4411. **Porous cup technique in the determination of magnesium in cast iron. A statistical study.** A. C. Ottolini (General Motors Corp., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 447–449.—Chemical methods for the determination of Mg in cast iron are lengthy and inaccurate. The spectrographic method described is rapid and the coeff. of variation (95 tests) was $\pm$1·83%. A graphite porous cup was used as the sample electrode, and synthetic standards containing Fe and Mg were used to establish the analytical curve. A series of tests established instrument repeatability, chemical errors due to sample preparation, day-to-day reproducibility, and total over-all deviation.
P. D. PARR-RICHARD

4412. **Spectrographic analysis of small amounts of rare-earth elements in alloy steel.** Shüh-Cho Li and Yü-Me Wang (Inst. of Metal Res., Acad. Sinica). *Acta Metallurg. Sinica*, 1959, **4** (1), 81–86.—In the method employed (*cf.* Spitz *et al.*, *Anal. Abstr.*, 1954, **1**, 1221), either the electrolytic separation with a mercury cathode or the ether extraction technique is used for the removal of interfering ions and for the concentration of rare-earth metals (*e.g.*, La, Ce, Pr and Nd) before the spectrographic procedure, Th being added as the internal standard. The concentrated sample is dissolved in a small amount of HCl (1:1). An aliquot (0·05 ml) is transferred to the end of the carbon electrode and dried at 250°. An a.c. arc (6 amp.; exposure 1 min.) is used to excite the spectra. The method permits the determination of as little as 50 μg of total rare-earth metals in a 1-g sample of steel with a max. error of $\pm$ 10%. S. H. YUEN

4413. **Quick determination of the phosphorus content in steel during manufacture, with the use of the isotope dilution method.** J. Fodor (Radioisotope Lab., Csepel Iron and Metal Factory, Budapest). *Acta Chim. Acad. Sci. Hung.*, 1959, **19** (1), 13–21 (in English).—The changes in phosphorus content of steel produced by the Siemens-Martin process are followed by introducing a drilled block containing a quantity of ^{32}P (0·1 mC per into the molten metal; the isotope is evenly di buted within 6 min. A sample is cooled to gi casting 9 mm thick and its β-activity is measu the test takes 5 min. and compares favourably chemical analysis for phosphorus contents o 0·005%. By employing standard conditions, e due to self absorption, etc. are kept constant initial absolute value can be obtained by cher means. P. D. PARR-RICHA

4414. **Rapid determination of niobium in stai steel.** R. St. J. Emery. *Metallurgia*, *Ma* 1959, **59**, 101–103.—The spectrophotometric cyanate method for determination of Nb has adapted to stainless steels. The separation removal of interfering elements is based on phase separation that occurs when the acid co of the extractant exceeds 1·4 moles per litre acetone as the organic medium. A 20% soln NH_4SCN, a 10% soln. of $SnCl_2$ and a dil. soln H_2SO_4 (1:1) are used and the extinction is rea 383 mμ. A determination can be made withi min. with satisfactory reproducibility.
J. W. O. PYEMO

4415. **Rapid method for the determinatio cobalt in reactor steel by activation analysis.** Westermark and I. Fineman (Royal Inst. of T Stockholm). Second U.N. International Confer on the Peaceful Uses of Atomic Energy, Gen 1958, Rep. A/CONF.15/P/140, 12 pp.— method is based on the use of the metast isomer ^{60m}Co which precedes the long-lived after neutron capture. The ^{60m}Co emits 59 γ-radiation and has only 10·8 min. half-life. enables a very short activation time to be ($\simeq$10 min. in the Stockholm reactor R1 at 5 $\times$ n per sq. cm per sec.). The method has been ap to various Swedish steels. The presence of M the most disturbing factor and has so far preve the use of the direct spectrometric analytical t nique (thick NaI crystal scintillation detectors a single-channel pulse-height analyser). A r procedure has been used to avoid this diffic It involves the rapid dissolution of steel turn in HCl, extraction of Fe into diethyl ether, selective pptn. (after addition of carrier) of Co nitrosonaphthol. The procedure also makes absorption of the soft γ-radiation negligible small activity of long-lived ^{60}Co is added to dissolving acid. This is chosen so as not to dis the spectrometry but to permit corrections losses of Co during the procedure. The stan consists of a small filter-paper on which a s volume of cobalt soln. is evaporated. The w procedure after activation occupies 30 to 40 and a sensitivity limit of 1 to 2 μg is achieved a neutron flux used. The estimated max. err 5 to 10% in the region 50 to 150 p.p.m. on a sample with the flux stated. NUCL. SCI. ABS

4416. **Solvent extraction method for biv cobalt.** S. Ionescu and C. Grigorescu-Sabău (de Phys. Atómique, Bucharest). Second International Conference on the Peaceful Use Atomic Energy, Geneva, 1958, Rep. A/CONF P/1280, 8 pp.—A comparative investigation o extraction of Co^{2+} and Co^{3+} in the presence of other in neutral soln. is reported. A more effi separation method for these ions is prop namely the extraction of Co^{2+} as $(Co^{2+}Py_4)(S$

HCl_3. The method can also be used with mounts. The formation of the complex was gated by this solvent extraction method each of the components in quantity, the eing in excess. In this way it was observed e radiometric titration with NH_4SCN can be successfully to the quant. determination of The equilibrium shift obtained during the n with pyridine allowed the calculation of emical potential for the combination of Co ridine to be made. NUCL. SCI. ABSTR.

Separation of cobalt from nickel by means n exchange and the photometric determination lt as the cyanate complex. M. Ziegler and tner (Anorg.-chem. Inst., Univ., Göttingen, ny). *Z. anal. Chem.*, 1959, **165** (3), 197–200. lumn (10 ml × 1 ml) of Dowex 1-X8 (50 to sh) retains 30 mg of Co as the cyanate com- t pH 6·5 to 8, while Ni passes through. *ure*—The sample containing ≯ 30 mg of Co 300 mg of Ni is neutralised with NaOH,) ml of KOCN soln. (20%) and 15 ml of ium acetate soln. (15%) are added. The pH 6·5 to 8) is then poured through the of Dowex 1-X8, and followed by 50 ml of The resin is removed from the column and eluted with 10 ml of HCl (20%). After isation with Na_2CO_3 soln. (20%), the Co evaporated to 30 ml and transferred to a flask; 10 ml of ammonium acetate and 40 ml CN soln. are added, and the soln. is made up The extinction is measured at 620 mμ.

B. B. BAUMINGER

Determination of small amounts of cobalt ium, zirconium and their alloys. D. E. Wood T. Clark (Res. Dept., I.C.I. Ltd., Metals Kynoch Works, Birmingham, England). *a*, 1959, **2** (1), 1–11.—Cobalt (2 to 50 p.p.m.) ium, zirconium and their alloys is determined tiometrically with nitroso-R salt at pH 6 presence of NH_4F. *Procedure*—Dissolve the (0·25 g) in H_2SO_4 (2% v/v) (10 ml) and soln. (1 ml). Warm to 70° to assist solution en add several drops of conc. HNO_3 and set or 5 min. Add NH_4F soln. (10%) (10 ml), boiling and add nitroso-R salt soln. (0·2%)) and ammonium acetate buffer soln. (50%)). Set aside for 5 min., add conc. HNO_3 and after 5 min. satd. bromine water (0·5 After a further 5 min. boil for 5 min., filter 50-ml flask, dilute to volume and measure the ion at 420 mμ in 4-cm cells. The method icable to titanium containing up to 20% of % of Al and 5% of Fe, and zirconium con- up to 5% of Pb, Al, Fe, Mn, Zn, Mg or W. mple modifications samples containing 20% or Mo, 5% of Cu, Cr or V, or 2·5% of Ni so be analysed. The standard deviation at m. is ≃ ±0·3 p.p.m. W. T. CARTER

Chelometric determination of cobalt and sing a fluorescent end-point. D. H. Wilkins Lab., General Electric Co., Schenectady, N.Y.,). *Talanta*, 1959, **2** (1), 12–15.—In EDTA ons of Fe and Co with visual indicators the weight is limited to ≃ 10 mg because of the colours of the complexes of Fe and Co with . This difficulty can be avoided by using a cent indicator. *Procedure*—To the slightly mple soln. containing up to 80 mg of Fe or d an excess of 0·03 *M* EDTA and adjust to pH 4·8 with Na acetate - acetic acid buffer soln. Dilute to 100 ml with water, add 1 drop of calcein soln. (0·1% in 0·001 *N* NaOH) and back-titrate with standard Cu soln. (0·03 *M*) under u.v. illumination. The end-point is detected by the quenching of the green fluorescence. W. T. CARTER

4420. **Determination of nickel, cobalt, iron and zinc in ferrites.** D. H. Wilkins (Gen. Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chim. Acta*, 1959, **20** (3), 271–273.—The four elements are separated by utilising the differences in adsorption of their chloride complexes on a strongly basic anion-exchange resin. *Procedure*—Dissolve the sample (1 g) in 9 *N* HCl (20 ml) and H_2O_2 (30%) (0·5 ml); transfer the soln. to the ion-exchange column (50 cm × 1 cm), and then elute the Ni with 9 *N* HCl (80 ml), the Co with 4 *N* HCl (75 ml), the Fe with 0·5 *N* HCl (100 ml), and finally the Zn with 3 *N* HNO_3 (100 ml). Evaporate each eluate to dryness and then determine each element by back-titration, at pH ≃ 4·8, of an excess (5 to 10 ml) of EDTA with 0·03 *M* $CuSO_4$, with 1-(2-pyridylazo)-2-naphthol as indicator. Results for Co and Fe are satisfactory, but those for Ni and Zn tend to be high. W. J. BAKER

4421. **Separation of nickel and cobalt by extraction with tributyl phosphate.** A. Musil and G. Weidmann (Inst. f. anorg. u. anal. Chem., Univ., Graz, Austria). *Mikrochim. Acta*, 1959, (3), 476–480.—Cobalt may be extracted from aq. soln. by means of tributyl phosphate (**I**). It is shown that even a threefold excess of Ni has little effect on the partition of Co between 8·3 *N* HCl and **I** - toluene (2:1). A method for the determination of Co in the presence of Ni is based on this finding.

D. F. PHILLIPS

4422. **Gravimetric determination of nickel with dimethylglyoxime in the presence of copper.** A. Claassen and L. Bastings (Philips Res. Lab., Eindhoven, Netherlands). *Z. anal. Chem.*, 1959, **165** (5), 354–360 (in English).—The interference of Cu is eliminated by pptn. of Ni with dimethylglyoxime in a soln. containing tartrate and $Na_2S_2O_3$ at pH 5·5 to 6·5. The amount of $Na_2S_2O_3$ added (minimum 3 g) is ≮ 15 times the weight of Cu plus Fe. Brass, bronze and steel are dissolved in HCl with the addition of HNO_3. With W-containing steel the ppt. of WO_3 dissolves when the soln. is made alkaline after the addition of tartaric acid. *Procedure*—To the sample is added 5 to 10 ml of 20% aq. tartaric acid soln., the mixture is made faintly ammoniacal with aq. NH_3, filtered if cloudy, and adjusted to pH 3 to 5 with dil. HCl. After dilution to about 200 ml the soln. is heated to ≯ 45°, then 15 ml of 20% aq. $Na_2S_2O_3$ soln., and, after a few minutes, the necessary amount of 1% ethanolic dimethylglyoxime soln. are added, followed by 25 ml of 2% aq. ammonium acetate soln. (pH 6·5 to 7·0). The mixture is stirred and cooled to room temp. for ≮ 30 min., and the ppt. is collected in a sintered glass crucible, washed with cold water and dried at 150°. Results for standard samples and synthetic mixtures are tabulated.

B. B. BAUMINGER

4423. **Photometric determination of nickel in copper - nickel alloys.** M. C. Steele and L. J. England (Metal Manufactures Ltd., Port Kembla, Australia). *Anal. Chim. Acta*, 1959, **20** (6), 555–558.—A rapid method is described for the determination

of from 1 to 32% of Ni. *Procedure*—Dissolve 1 g of the sample in 10 ml of conc. HNO_3 and boil off the nitrous fumes. Allow to cool to 80°, add 10 ml of H_3PO_4, cool to room temp. and dilute to 25 ml. Filter if necessary and measure the transmittancy at 390 mμ in a Beckman model B spectrophotometer with 2-cm cells, against a reference soln. containing approx. 1% less than the expected content of Ni. The calibration graph is prepared with soln. in which the total of Cu plus Ni is 1 g, and determinations and calibration must be conducted at the same temp. ($\pm 1°$). None of the elements likely to be present interferes, but the addition of Cl^- must be avoided. H. N. S.

4424. **Critical review of the volumetric methods for the platinum metals.** F. E. Beamish (Univ. of Toronto, Ontario, Canada). *Anal. Chim. Acta*, 1959, **20** (2), 101–112.—The review covers methods for Pd, Pt, Rh, Ir, Ru and Os published up to Jan., 1958. Attention is drawn to the lack of methods generally and directly applicable to the analysis of ores, concentrates and alloys. (61 references.) H. N. S.

4425. **Quinolinic acid as a reagent for the gravimetric and volumetric estimation of palladium.** A. K. Majumdar and S. P. Bag (Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1959, **165** (4), 247–250 (in English).—Quinolinic acid forms a complex with Pd of composition $Pd(C_7H_4O_4N)_2$, which is pptd. at an acidity of 0·25 *N* with respect to HCl or HNO_3, up to a max. pH of 2·1. Most common ions and the other platinum metals remain in soln. The only interfering ion, Cu, is eliminated by complexing with EDTA. The Pd complex is either determined gravimetrically or dissolved in standard KCN soln., which is back-titrated with $AgNO_3$ soln. Recoveries are >99% by both procedures. G. P. Cook

4426. **Separations involving palladium. Separation of lead, bismuth, cadmium, thallium, copper, cobalt and nickel from palladium.** G. B. S. Salaria (Gov. Coll., Rohtak, Punjab, India). *Anal. Chim. Acta*, 1959, **20** (2), 122–123.—A soln. of $PdCl_2$ (45 to 58 mg of Pd) and Pb acetate (49 to 62 mg of Pb) in 2 *N* to 3 *N* HCl is treated with 20 ml of a 1% alcoholic soln. of dimethylglyoxime (**I**) and is set aside for 1 hr. with occasional shaking. The Pd complex is filtered off in a sintered glass crucible (porosity 4), washed with hot water acidified with HCl, then with alcohol, dried at 110° and weighed. The same procedure serves for the separation of Pd from Bi, Cd, Tl, Cu, Co and Ni. In the filtrates, Pb, Bi and Cd are determined as their sulphides, Tl as Tl_2CrO_4, Cu by thiosulphate titration, Co as the pyridine complex, and Ni with **I**. H. N. S.

4427. **Thermogravimetry of analytical precipitates. LXV. Determination of iridium.** C. Duval, P. Champ and P. Fauconnier (École Nat. Supérieure de Chim., Paris, France). *Anal. Chim. Acta*, 1959, **20** (2), 152–153 (in French).—Iridium, pptd. as the metal with formic acid, yields a horizontal thermogravimetric graph up to 879°, showing that heating in hydrogen is unnecessary. The ppt. obtained with 2-mercaptobenzothiazole shows an inflection in the curve at 135° to 250°, and a horizontal portion from 520° to 980° indicates conversion into the metallic state. The ppt. obtained with Na_2S gives a constant wt. only after the free metal is formed at $\simeq$800°. H. N. S.

4428. **New and improved methods of analys some nuclear raw materials.** V. T. Athaval Banerjee, G. K. Belekar, N. Mahadevan, L Mahajan, M. N. Nadkarni, Das M. Sankar, F Sharma, A. K. Sundaram, M. Sundaresan, N Thakoor, M. M. Tillu, M. S. Varde and C. kateswarlu (Atomic Energy Estab., Trom India). Second U.N. International Conferenc the Peaceful Uses of Atomic Energy, Geneva, Rep. A/CONF.15/P/1671, 15 pp.—A brief re is presented of the new and improved met developed in the Trombay laboratory during the few years for the analysis of minerals and ore U, Th, Be, Nb and Ta present in macro and r amounts. Uranium and Th determinations also required in uranium and thorium cr intermediates, and pure thorium nitrate sampl Nucl. Sci. Abs

4429. **Testing of nuclear fuels by the da coefficient method.** M. E. Remley and B Engholm (Atomics International Div., N. Amer Aviation, Inc., Canoga Park, Calif.). C "Modern Approaches to the Isotopic Analys Uranium." Chicago, Feb., 5–7, 1957. Rep. 7531(Pt.2)(Del.), 53–69.—Reactors operating essentially zero power can be used as sens non-destructive test-instruments for quality co and isotopic assay determinations of nuclear f The use of a zero-power water boiler for this of testing has been investigated in a series of ex ments performed with the Water Boiler Neu Source at Atomics International. Uranium inders, 3·875 in. in length by 0·500, 0·600 and in. in diam., with enrichments in ^{235}U of 0 0·711, 0·902, 1·399 and 2·778% by weight, been compared by the danger coeff. method. method with the water boiler will detect enrichment differences of 9×10^{-4} % by w for samples enriched to 1% in ^{235}U and differ of $2{\cdot}8 \times 10^{-3}$ % by weight in samples of enrichment. A suggested procedure for the a cation of the method to the routine testing of materials is outlined. Nucl. Sci. Abs

4430. **Analysis of Italian uraniferous mine I. Leaching with dilute sulphuric acid and co tration on exchange resins.** M. L. Borlera (Pol Turin, Italy). *Ric. Sci.*, 1958, **28** (2), 331–3 The minerals, an Italian autunite and an It pitchblende, are leached with boiling *N* H_2 The combined extracts are adjusted to pH 1 2·1 and passed through a strong anion-exch resin (Amberlite IRA-410), $UO_2(SO_4)_2^{2-}$ retained and then eluted with 0·25 to 0·5 *N* ($HClO_4$, HNO_3 or HCl). Nitric acid is prefe when phosphate is to be separated from the el Recovery of U of 98 to 99% from the leaching is obtained. A 5-fold concn. of U in the e relative to that of the soln. entering the res obtained. Most of the phosphate accompanie U. The Fe^{3+} are reduced to Fe^{2+} with SC avoid exchange of ferric sulphate anions. manganate cannot be eluted and thus may dim the capacity of the resin. Chloride and ni decrease the exchangeability of the uranyl phate. Chem. Abs

4431. **Identification of minerals associated asbestos by X-ray diffraction patterns.** M Badollet and J. P. McGourty (Johns-Manville Center, Manville, N.J.). *Canad. Min. M Bull.*, **51**, 335–340; *Trans. Canad. Inst. Metall.*, 1958, **61**, 169–174.—Serpentine, chrys antigorite, brucite, iron oxide, grammatite, ep

uartz were photographed by a Norelco X-ray tion unit with a Debye - Scherrer camera for exposure by copper radiation with a nickel This investigation indicates that for the ete identification of all the minerals present ples obtained in prospecting or in drill-cores estos bodies, not only chemical analyses but e results of X-ray diffraction, spectrographic, etrographic studies are necessary.

CHEM. ABSTR.

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also Abstracts—**4239**, Use of Zn toluene-3:4- . **4240**, Dithio-oxamide in detection of . **4241**, Use of sulphathiazole in inorganic is. **4242**, Gallein as reagent for Pb. **4246**, ation of bivalent from tervalent ions in the ce of PO_4^{3-}. **4252**, Photometric determina- of end-points in complexometric titrations. Extraction in inorganic analysis. **4260**, ery of trace elements from organic materials. Determination of Hg in paper, **4507**, Strontium ne by X-ray fluorescence. **4605**, Determina- f U in water. **4607**, Determination of Ra-C h-C in springs. **4608**, Determination of Ra-B Th-B in springs. **4628**, Isotopic analysis by erential spectrometry. **4647**, Determination and Ti. **4649**, Determination of Co.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives.

2. The bromimetric determination of organic ounds. A. Berka and J. Zýka (Dept. Anal. , Charles' Univ., Prague). *Českosl. Farm.*, **8** (1), 17–18.—It has been shown that the etric titration of the excess of reagent in ct bromimetric determinations (*e.g.*, of Na late and resorcinol) can be replaced by a on with hydrazine sulphate. *Procedure*— ve the sample (0·2 to 0·5 g) in H_2O (100 ml). ml of this soln. add 0·1 *N* $KBrO_3$ (25·0 ml), (1 g) and dil. HCl (5 ml). After 10 min. e potentiometrically with 0·05 *M* hydrazine ate (**I**), or add an excess of **I** and methyl red 3 drops) as indicator and back-titrate with $KBrO_3$ to a colourless end-point. The results in good agreement with the iodimetric back- on of the excess of $KBrO_3$ according to Cz.P.2.

J. ZÝKA

3. Catalytic hydrogenation of organic com- s on paper for paper-chromatographic analysis. . Kaufmann and Dipti Kalyan Chowdhury n. Inst. Fettforschung, Munster, Germany). *Ber.*, 1958, **91**, 2117–2121.—The appropriate lic hydrogenation catalyst, such as Pd, is ced in finely dispersed form on paper, the ound to be analysed (*e.g.*, unsaturated fatty nitro compounds, nitriles, dyes) is applied to etal spot, reduced in an atmosphere of hydro- nd chromatographed in the usual way. A 5% soln. in 20% HCl is applied once or several in amounts of 15 to 20 μg of Pd to both sides chromatographic paper at the starting line, and dried at room temp. The paper is treated at the same places on both sides with small drops of a mixture of 80 ml of 20% aq. KOH soln. and 20 ml of 40% aq. formaldehyde soln. in such a manner that the resulting black spots due to Pd do not become larger than about 1 cm, and is then immersed for 10 min. in 5% aq. acetic acid, washed 5 or 6 times with water, pressed between filter-papers and dried at 90°. This gives the palladium paper (*A*). *A* is impregnated with undecane (previously saturated with acetic acid) and dried for 10 min. in air. A mixture of oleic (**I**), linoleic (**II**) and linolenic (**III**) acids in toluene is applied to the middle of the palladium spot and also at some distance on the base line. The paper, after being placed in a vacuum desiccator, kept for 5 min. under hydrogen, and developed with 95% acetic acid, gave 1 spot for stearic acid from the sample applied to the palladium spot and 3 spots for **I**, **II** and **III** from the palladium-free spot. The method has been applied to linseed oil. Hydrogenation reduces the spot due to oleic plus palmitic acids to one due to palmitic acid alone. The palmitic acid can thus be determined. The method has also been applied to a fish oil, sorbic acid, maleic acid, nitrobenzene, *m*-dinitrobenzene, phenyl cyanide, indigo carmine and KIO_3.

CHEM. ABSTR.

4434. **Combustion of organic compounds in an oxygen atmosphere.** J. Horáček (Chem. Inst., Acad. Sci., Prague). *Chem. Listy*, 1959, **53** (1), 6–9.—A survey is given of the history and extension of the Schöniger method (*Mikrochim. Acta*, 1955, 123; 1956, 869) for the determination of S in organic compounds after the combustion of the sample in an atmosphere of O. The method is considered to be reliable, simple and precise, and suitable for micro-determinations. This method can be applied to the determination of other elements (*e.g.*, F, Br, Cl, P, Hg and ^{14}C).

J. ZÝKA

4435. **Determination of active hydrogen in organic compounds by exchange with tritiated *iso*propyl alcohol.** J. F. Eastham and V. F. Raaen (Univ. Tennessee, Knoxville, U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 555–558.—The active hydrogen in a variety of organic compounds is exchanged for tritium from an excess of tritiated *iso*propyl alcohol by dissolving each compound in the alcohol and then evaporating the solvent. The measured activity acquired by most compounds agrees well with that expected from the number of active hydrogen atoms in the compounds, and the known radioactivity of the solvent. Data for **33** compounds are listed.

G. P. COOK

4436. **Determination of carbon dioxide in the wet combustion of organic substances by the equilibration of pressures method.** V. A. Semin'ko. *Trudy Khar'kovsk. Farmatsevt. Inst.*, 1957, (1), 158–159; *Ref. Zhur., Khim.*, 1959, (2), Abstr. No. 4376.—A method based on the equilibration of pressures is described for the determination of the CO_2 liberated in the oxidation of organic substances in a closed flask. A volumetric manometer to correct automatically for changes in temperature and atmospheric pressure is illustrated, and comprises a manometer with an equalising bulb and a compensating chamber. The flask with the sample and the oxidising mixture is placed near the volumetric manometer and the pressure in the compensating chamber is adjusted to atmospheric. After the combustion, the flask is connected with the manometer (the vol. of gas in the flask being increased by

the CO_2 evolved), and after equalising the pressures the amount of CO_2 is calculated by the formulae given. The error of the determination is $\pm 1\%$.
C. D. KOPKIN

4437. **Organic micro-analysis. XX. Modified Dumas nitrogen determination: the correct temperature for the reduced copper.** Keiichiro Hozuma and Sachiko Amako (Pharm. Inst., Med. Fac., Univ. of Kyoto, Japan). *Mikrochim. Acta*, 1959, (2), 230–242 (in English).—Apparatus and procedure are described in detail. Experiments show that the temp. of the reduced copper in the combustion tube should be maintained at 550° to 600°, in which range complete reduction of nitric oxide is attained. Above 650°, large errors due to formation of CO were observed, and, below 500°, the reduction of nitrogen oxides is incomplete.
B. B. BAUMINGER

4438. **Detection and identification of halides in organic compounds. An improved micro qualitative procedure using zinc - lithium carbonate as fusion mixture.** E. E. Gansel (Anal. Sect., Res. Dept., Ansco, Div. of GAF, Binghampton, N.Y., U.S.A.). *Microchem. J.*, 1959, **3** (1), 91–95.—The sample (3 to 5 mg) is intimately mixed with 25 mg of the fusion mixture (a 1:1 mixture of zinc dust and Li_2CO_3) in a heavy-walled Pyrex-glass fusion tube (75 mm × 13 mm) and a further 50 mg is placed on top. A glass-wool plug is tamped down on to the mixture, and 0·5 ml of diethyl ether added to expel air. The tube is heated to a white heat in an oxy-gas flame, commencing at the top and taking care to avoid puncture of the tube by the fused mass. The tube is then dropped into a lipped Pyrex-glass test-tube (115 mm × 26 mm) which contains 5 ml of ice-cold water and a short length of Pyrex-glass tube (17 mm × 10 mm) to break the fall and keep the hot fusion-tube from the walls of the test-tube, a cover being placed over the tube to avoid spattering. The digestion tube is swirled for 1 to 2 min., the contents are boiled and filtered, and the residue is extracted with a further 2 ml of water. The combined filtrates are then tested for halogens in the usual way. The advantages claimed are lower melting-point of the fusion mixture and low solubility of Li_2CO_3 in water. R. E. ESSERY

4439. **Rapid micro-determination of fluorine in organic compounds.** R. N. Rogers and S. K. Yasuda (Univ. Calif., Los Alamos, U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 616–617.—The sample is decomposed by combustion in a flask filled with oxygen at atm. pressure and the fluorine determined by an improved ferric salicylate colorimetric method. The results are generally within 3% of those expected from theory and the analysis time is 10 to 20 min. for samples of from 0·4 to 20 mg.
G. P. COOK

4440. **Reduction of organic fluoro compounds with lithium and *n*-propylamine in the micro-determination of fluorine.** R. D. Strahm (Redstone Arsenal Res. Div., Rohm & Haas Co., Huntsville, Ala., U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 615–616.—Organic fluoro compounds are decomposed with Li and *n*-propylamine. The fluoride is separated from the residue by steam-distillation with $HClO_4$ and is determined by back-titration with $Th(NO_3)_4$ in Nessler tubes. Results are within 2% of the theoretical. G. P. COOK

4441. **Semi-micro determination of fluorine and chlorine in organic compounds. III. The decomposition of fluoro and chlorofluoro compounds by the diphenyl - sodium - dimethoxyethane complex.** Johncock, W. K. R. Musgrave and A. Wiper (I of Chem., Univ., South Road, Durham, Engl *Analyst*, 1959, **84**, 245–247.—The organic comp (≡≃8 mg of total halogen) is dissolved in 2 of di*iso*propyl ether, 10 ml of a 1 to 1·5 *M* sol the diphenyl - sodium - dimethoxyethane com (Liggett, *Anal. Chem.*, 1954, **26**, 269) is added the mixture is set aside for 10 min., with occas swirling. The liquid is extracted with w (3 × 20 ml), the combined aq. extracts are adju to 100 ml and a 25-ml aliquot is placed on a col of Amberlite IR-120(H). The column is el with 70 ml of boiled water, the eluate is heate boiling, cooled immediately and titrated with 0· NaOH, with screened methyl red as indic When more than one halogen is present the cedure of Banks *et al.* (*Anal. Abstr.*, 1956, **3**, 1 is followed. A. O. JON

4442. **New and rapid micro-analytical determ tion of chlorine, bromine and fluorine in org compounds.** F. Ehrenberger (Anal. Lab., F werke Hoechst, A.-G., Frankfurt, Germa *Mikrochim. Acta*, 1959, (2), 192–201 (in Germa The organic compound is vaporised in a strea H and burnt in an oxy-hydrogen flame. quartz combustion tube and absorber are illustra Chlorine and Br are determined potentiometri with 0·02 *N* $AgNO_3$ and details of the titratio fluorine with 0·01 *N* $Th(NO_3)_4$ are given. A and alkaline-earth metals interfere in the d mination of halogens, and phosphate must be moved with $ZnCO_3$ before titrating with Th(N soln. B. B. BAUMING

4443. **Analysis for industry.** A. M. G. Macdo *Ind. Chem.*, 1959, **35**, 33–35, 37.—A revie presented of work on the combustion of org samples by the closed-flask technique, and app tions to the determination of Hg, S, P, ^{14}C, Ca, Cd and B are described. (26 references.)
S.C.I. ABST

4444. **Micro-determination of the carbonyl g by oxime formation.** V. A. Klimova and K Zabrodina (Inst. Org. Chem., Acad. Sci., SSSR). *Akad. Nauk SSSR*, 1959, (1), 175–176.—*Procedu* Dissolve hydroxyammonium chloride (0·7 g H_2O, add triethanolamine (0·5 g) and make u 100 ml with 96% ethanol. Shake the test subst (5 to 10 mg) with this soln. (5 ml) until it diss and then set aside for 30 min. at room temp. satd. NaCl soln. (2 ml) and bromophenol blu drops) to the control and titrate it with 0· HCl. Titrate the test soln. in the same way to the same visual end-point. The results fo analyses of aldehydes and ketones are tabul Ketones that possess methylene groups with hydrogen atoms adjacent to the carbonyl group be analysed by this method. K. R.

4445. **Application of diphenylacraldehyde to f tional analysis.** M. Pesez and J. Bartos (Ser Scientifiques Roussel-Uclaf, Paris, France). *Chim. Acta*, 1959, **20** (2), 187–189 (in Frenc When 2 ml of a 1% methanolic soln. of **3**: phenylacraldehyde (**I**) and 1 ml of conc. HC added to 2 ml of a methanolic soln. of a *m*-dih phenol a red colour is produced. The reactio sensitive to 1 μg of the phenol and the co intensity follows Beer's law for 2 to 10-μg amo Under the same conditions **I** condenses with c ketones giving yellow to yellow - green or colours. The products of condensation *cyclo*hexanone and barbituric acid have

and structures have been attributed to Schiff's bases are formed when **I** reacts with aromatic amines, and the orange - yellow comply with Beer's law for quantities of p to 20 μg. H. N. S.

New method for the determination of groups. L. Mázor and T. Meisel (Tech. Budapest, Hungary). *Anal. Chim. Acta*, (2), 130–133 (in German).—A rapid method bed for the determination of acetyl groups tyl compounds. The sample (45 to 300 mg) ved or suspended in methanol and is saponi- 1 10 ml of 0·1 *N* K methoxide. The follow- tions take place—

$$CH{\cdot}O{\cdot}CO{\cdot}CH_3 + CH_3OK \rightarrow >CH{\cdot}O{\cdot}C(CH_3)(OCH_3)(OK)$$

$$CH{\cdot}O{\cdot}C(CH_3)(OCH_3)(OK) + CH_3OH \rightarrow >CH{\cdot}OH + CH_3{\cdot}CO{\cdot}OCH_3 + CH_3OK.$$

s then added to hydrolyse the K methoxide. OH so formed hydrolyses the methyl acetate excess of KOH is titrated with 0·1 *N* HCl, enolphthalein as indicator. If the reaction is coloured, a fluorescent indicator is used, ethyl acetate is distilled after the completion ions (*i*) and (*ii*) and the ester is hydrolysed distillate with an excess of 0·1 *N* NaOH. thod is applied to the acetates of several to hydroxypregnenone acetate, 3α-acetoxy- st-22-ene, cellulose triacetate, diacetylmor- 2-naphthyl acetate and poly(vinyl acetate). H. N. S.

Partial automation of acetyl determination: tic control of the acetic acid distillation. nz (II Chem. Inst., Univ. Wien, Austria). . *Chem.*, 1959, **166** (1), 32–36.—The most stage in the determination of acetyl groups quant. distillation of acetic acid from aq. ntaining H_2SO_4 which involves the repeated 0) additions of small quantities of H_2O. A for making these additions automatically es a pump controlled by a relay, actuated electrodes which operate when the liquid distillation flask falls below a given level. ctrodes are set to maintain a vol. of about the distillation flask. The distillation take 20 to 30 min. and yield 50 to 60 ml of e. A modified Kjeldahl apparatus for the e determination is described. G. Burger

Ultra-violet absorption in solvent analysis. mprecht (Inst. für Lackforsch., Giessen). *Seif., Anstrichmitt.*, 1959, **61** (2), 96–99.— plication of u.v. absorption measurements to lysis of solvents is considered. Absorption for compounds typical of various solvent *e.g.*, aromatics, chlorinated hydrocarbons, ketones and alcohols), and certain of their mixtures, are shown and discussed. It is ed that u.v. spectral analysis can be a useful al tool in the analysis of solvents, par- y when the complete separation of com- s is not possible by other methods. Quanti- analysis may also be possible by using the ence of extinction on concn. S. M. Marsh

Separation of C_2 to C_5 hydrocarbons by gas-partition chromatography. N. M. Turkel'taub, nvaer, A. I. Kolyubyakina and M. S. Selen- All-Union Sci. Res. Geological Prospecting um Inst.). *Zavod. Lab.*, 1959, **25** (2), 149– ncomplete separations of C_4 and C_5 isomers, even with optimum ratios between adsorbents and solvents, are reported. Mixed solvents, *e.g.*, dimethylformamide containing 1% of liquid paraffin on crushed brick, or dimethylformamide containing 6·5% of tri*iso*butylene on diatomite, can give complete separations of C_4 and C_5 hydrocarbons, and it is considered that conditions can be chosen for separating C_2 to C_5 hydrocarbons. G. S. Smith

4450. **Chromatographic analysis of mixtures of C_5 hydrocarbons.** A. A. Zhukhovitskiĭ, B. A. Kazanskiĭ, O. D. Sterligov and N. M. Turkel'taub (Akad. Nauk SSSR, Moscow). *Dokl. Akad. Nauk SSSR*, 1958, **123** (6), 1037–1040.—Analytical methods for the products of the catalytic dehydrogenation of *iso*pentane into mixtures of *iso*amylenes and isoprene were investigated. The universal chromathermograph method was most convenient and the accurate calibration of chromatograms of synthetic mixtures of up to ten saturated and unsaturated C_5 hydrocarbons enabled this instrument to be used for the analysis of complex mixtures of C_5 hydrocarbons. The results were all within 3% of the correct values and the instrument was utilised for purity determinations. A. Grochowski

4451. **Gas chromatography of hydrocarbons using capillary columns and ionisation detectors.** A. Zlatkis and J. E. Lovelock (Dept. of Chem., Univ. of Houston, Tex., U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 620–621.—A 100-ft. copper capillary-column, 0·01 in. in diam., coated internally with squalane and with A as carrier gas, has been used successfully in conjunction with an ionisation detector to resolve all the C_5 to C_7 paraffins, *p*- and *m*-xylene, all the C_1 to C_5 alcohols and aq. soln. of C_2 to C_5 aldehydes. The max. load appears to be 1 μg of a mixture containing 10 components, and an efficiency of 750 theoretical plates per ft. has been reached in the separation of hydrocarbon mixtures. K. A. Proctor

4452. **Determination of acetylene in ethylene oxide.** H. L. Spell (Dow Chem. Co., Freeport, Tex.). *Anal. Chem.*, 1959, **31** (8), 1442.—Infra-red data are given.

4453. **Infra-red analysis of monochloropropene mixtures.** H. L. Spell (Dow Chem. Co., Freeport, Tex.). *Anal. Chem.*, 1959, **31** (8), 1442.—Infra-red data are given.

4454. **Determination of ethyl bromide by means of electro-reduction.** E. G. Maleeva. *Nauch. Dokl., Vŷssh. Shkol. Khim. i Khim. Tekhnol.*, 1958, (3), 505–506; *Ref. Zhur., Khim.*, 1959, (5), Abstr. No. 15,117.—A small amount of ethyl bromide (0·1 mg) is determined in a special electrolyser with platinum or nickel electrodes 4 cm apart. Reduction is carried out at 37° and with a current of 400 mA in an ethanolic medium containing 10% of alkali; the test takes 20 min. In the reaction mixture Br^- are determined argentimetrically with an adsorption indicator. K. R. C.

4455. **Determination of tetrabromoethane and mineral acids dissolved therein.** M. Rogozinski and L. M. Shorr (Israel Mining Industries, Haifa). *Analyst*, 1959, **84**, 66–67.—The assay is based on the ease with which tetrabromoethane (**I**) is converted by alkali into $CHBr:CBr_2$ and alkali bromide. The sample (>150 mg of **I**) is heated to b.p. with 20 to 30 ml of methanol and titrated with 0·1 *N* NaOH to phenolphthalein indicator. When the

red colour appears, the liquid is boiled again and titration is continued until the colour is permanent after boiling for 10 sec. The determination can be checked by slight acidification of the titrated soln. with 0·1 *N* acetic acid and titration of the alkali bromide with 0·1 *N* $AgNO_3$, with dichlorofluorescein as indicator. The dehydrohalogenation equiv. is [wt. (g) of sample × 10,000]/vol. (ml) of 0·1 *N* NaOH. For amounts of **I** from 2 to 150 mg, 0·01 *N* reagents are used, the titration with alkali is taken beyond the end-point, to ensure complete dehydrohalogenation, the liquid is neutralised with 0·01 *N* acetic acid and the alkali bromide is titrated with 0·01 *N* $AgNO_3$. If the original sample contains HBr, excess of KIO_4 and KI are added to its soln. in methanol and the liberated iodine is titrated with $Na_2S_2O_3$ soln. A. O. Jones

4456. **Detection and measurement of hydroperoxides by near infra-red spectrophotometry.** R. T. Holman, C. Nickell, O. S. Privett and P. R. Edmondson (Hormel Inst., Univ. of Minnesota, Austin, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1958, **35** (8), 422–425.—Hydroperoxides formed by autoxidation are characterised by absorption maxima at 1·46 and 2·07 μ; the intensity of absorption is proportional to the peroxide content. The effect is not shown by dialkyl peroxides or ozonised unsaturated substances. The spectral detection of –OOH is not possible when the peroxide value is <500 milli-equiv. per kg.
P. D. Parr-Richard

4457. **Organic chemical microscopy. IV. 3:5-Dinitrobenzoates of hydroxy compounds in qualitative organic analysis.** R. E. Dunbar and F. J. Ferrin (North Dakota State Coll., Fargo, U.S.A.). *Microchem. J.*, 1959, **3** (1), 65–82.—Alcohols, glycols, phenols and related compounds are purified, and 1 ml of a liquid, or 0·5 g of a solid dissolved in diethyl ether, is treated with metallic sodium till no further reaction is observed. If the reaction mixture is solid, a little of the original liquid, or of ether, is added to give a semi-liquid mass, to which 0·15 g of 3:5-dinitrobenzoyl chloride is added, with stirring and cooling if necessary. Water (10 ml) is added and the whole allowed to crystallise. The crystals are filtered off, triturated with 10 ml of 2% aq. Na_2CO_3 soln. and re-filtered. The crystals are dissolved in acetone and allowed to crystallise after filtering. (When only oily drops are formed, the mixture is extracted with ether, the ether evaporated, and the residue crystallised from ethanol, but rapid manipulation is necessary to avoid esterification.) The derivatives are recrystallised from acetone and examined at a magnification of × 150. Photographs are given of derivatives from 40 single compounds, 8 binary and 1 ternary mixture. The results are discussed.
R. E. Essery

4458. **Effect of sodium borate on the optical rotation of sugars.** R. C. Hughes and W. J. Whelan (Lister Inst. of Preventive Med., London). *Chem. & Ind.*, 1959, (2), 50.—The changes in optical rotation which occur in 0·6 *M* borate buffer at pH 10 are listed for 29 mono-, di- and tri-saccharides.
K. A. Proctor

4459. **Separation and determination of isomaltose and gentiobiose.** Nobuko Seno (Zool. Lab., Fac. Sci., Ochanomizu Univ., Tokyo). *Nat. Sci. Rep., Ochanomizu Univ.*, 1958, **9** (2), 57–60.—Paper chromatography and paper electrophoresis have been used to identify isomaltose (α-1:6) and gentiobiose (β-1:6). Electrophoresis on Whatman paper in a borate buffer (pH 9·8) is more s than paper chromatography. For their determination, ion-exchange chromatograp employed using the borate complexes on a base anion exchanger (Dowex 1). (17 refer (From English summary.) S.C.I. Ab

4460. **A remark on the refractive-index inc of dextran for the molecular-weight determi by light scattering.** M. Zebec, G. Deželić, J. K vil and K. F. Schulz (Dept. of Phys. Bio "Andrija Štampar" Sch. of Public Health, U Zagreb, Yugoslavia). *Croat. Chem. Acta*, **30** (4), 251–255 (in English).—Values have determined for the refractive-index change concn. (dn/dc) for aq. dextran soln. at 4 546 mμ and 578 mμ. The dextran was dis by pouring the powder into boiling wate heating for 2 hr. in a water bath at 100°. concn. of the soln. was found by drying an a in a vacuum oven over P_2O_5 at 110°. $\triangle n$ was ured for a series of soln. of various concn. by of an interferometer, the wavelength being se with a mercury vapour lamp and suitable The values found for dn/dc (cu. cm per gram) at 436 mμ, 0·1518 ± 0·0012; at 546 mμ, 0·1 0·0013; at 578 mμ, 0·1476 ± 0·0013. H

4461. **Analysis of sodium formaldehyde-bisu and sodium formaldehydesulphoxylate (rong** L. Maros (Inst. f. Anorg. u. Anal. Chem., L. E Univ., Budapest). *Acta Chim. Acad. Sci.* 1959, **19** (1), 57–64 (in German).—This is a G version of the article published original Hungarian in *Magyar Kém. Foly.*, 1958, ((*cf. Anal. Abstr.*, 1958, **5**, 3779).
P. D. Parr-Rich

4462. **Quantitative determination of trace an of carbonyl compounds. II. Colorimetric a for acetaldehyde.** E. W. Malmberg, B. Wei D. L. Fishel and R. A. Krause (Dept. of C Ohio State Univ., Columbus, U.S.A.). *Mikr Acta*, 1959, (2), 210–213 (in English).—The m is based on the addition of 2-(4-phenylazo)p hydrazinesulphonic acid to the strongly acid aldehyde soln. and extraction of the lav coloured product with $CHCl_3$ in the prese ethanol. Procedures for concn. of 25 μg p and of 1 to 5 μg per ml of aldehyde are desc The aldehyde can be determined with an acc within ± 0·5 p.p.m. B. B. Baumin

4463. **General survey of the characterisatio determination of acids and acid anhydride** Veibel (Tech. Univ., Copenhagen, Den *Chim. Anal.*, 1959, **41** (1), 12–17.—A lite survey is presented, and procedures are outlin the identification of acids by the formation of esters, amides and anilides, the identificati amino acids, the use of partition chromatog the identification of acid anhydrides, an detection and identification of acids and al combined as esters. Forty-nine reference numbered but not otherwise identified.
R. E. Ess

4464. **Micro-titration of weak organic ac non-aqueous solvents.** R. K. Maurmeye Margosis and T. S. Ma (Dept. of Chem., Bro Coll., New York City, U.S.A.). *Mikrochim.* 1959, (2), 177–191 (in English).—The tit vessel (50 ml) has five necks, one in line wi vertical axis of the flask and the other four e

and slightly oblique. The vertical neck has joint for the micro-burette, and two dially opposed side openings are used for des. A stream of N passes through the wo openings to minimise interference by CO_2. titrations in dimethylformamide (**I**), pyridine ethylenediamine and 3-methylbutan-2-one vith thymol blue and azo violet as indicators, cribed. **II** is used for the titration of organic hat are too weak to be titrated accurately .g., sulphanilamide). Visual titrations in **I** ployed for compounds with pK_a values of d potentiometric titrations for those with f $\simeq$10·5. With thymol blue as indicator, unds with a pK_a of $\simeq$9 can be titrated in **III**.
B. B. Bauminger

. **Behaviour of acidic organic compounds in ueous media on ion-exchange resins.** R. N. and C. J. Umberger (Hosp. for Special y, New York, U.S.A.). *Anal. Chem.*, 1959, Part I), 593–595.—Compounds of different are adsorbed on strongly basic ion-exchange and eluted with ethanol or acidic ethanol; the ual fractions are identified by u.v. spectrometry. Recoveries were > 93% for the 20 unds tested. G. P. Cook

. **Titration of mixtures of amines in a non-s medium.** E. A. Gribova and É. S. Levin Voroshilov Inst. of Org. Intermediates and ffs). *Zavod. Lab.*, 1959, **25** (1), 38–41.—The iometric titration with 0·1 *N* $HClO_4$ in dioxan acetone soln. of a mixture of *cyclo*- and di-exylamines and aniline gives two potential the first corresponding to the content of exylamines and the second to that of aniline. ylaldehyde is first added, the single potential obtained corresponds to the content of di-exylamine alone. Hence from the results of rations the amounts of all three components e determined. G. S. Smith

. ***p*-Chlorobenzoyl *iso*thiocyanate as reagent haracterisation of primary and secondary s.** M. Tišler (Univ. of Ljubljana, Yugoslavia). *l. Chem.*, 1959, **165** (4), 272–275.—*p*-Chloro-yl *iso*thiocyanate reacts almost quant. with ry and secondary amines in the cold. The tives are re-crystallised from ethanol and are ied by their m.p. Data for $\simeq$50 amines are
G. P. Cook

. **Identification of secondary β-hydroxyethyl-s by fluorescence, polarography, paper chrom-phy and thermochromy.** K.-H. König and rg (Inst. f. Mikrobiol. und exper. Therapie, Germany). *Z. anal. Chem.*, 1959, **166** (2), .—Compounds of the general formula)·CH_2·CH_2·OH can be identified by reaction excess of *p*-benzoquinone (or preferably luquinone) in ethanol or acetone; the prod-2-substituted quinones) are formed rapidly have characteristically. They are separated 2; 5-di-substituted by-products by paper-disc atography, and give a yellow zone strongly cent in u.v. light. Brief warming changes llow to violet (thermochromic change) and ange is reversible (but not after prolonged ng). On polarography the substances show racteristic two-electron equilibrium doublet. way 10^{-5} to 10^{-6} g of hydroxy-amine can be ied. Primary 2-hydroxyethylamines give -substituted benzoquinones only; tertiary oxyethylamines do not react. J. P. Stern

4469. **Determination of small amounts of tri-*iso*octylamine in aqueous solution.** A. W. Ashbrook (Res. and Devel. Div., Eldorado Mining and Refining Ltd., Ottawa, Ontario, Canada). *Analyst*, 1959, **84**, 177–179.—The method was developed for the determination of tri*iso*octylamine (**I**) in aq. soln. resulting from the extraction of uranium-bearing ores. To the sample soln. NaH_2PO_4 is added [to prevent formation of $Fe(SCN)_3$] and 20 ml of a soln. of $Co(NO_3)_2$ and NaSCN (10 g of each in 100 ml of water) and the mixture is shaken. It is then extracted with a known vol. of CCl_4 and the extinction of the separated and filtered organic layer is measured at 620 mμ and referred to a calibration graph. The standard soln. for calibration are prepared by dissolving 0·5 g of **I** in 1 litre of methanol, acidifying portions of this soln. with 2·5% H_2SO_4 (10 ml), diluting to 80 ml and developing and extracting the Co complex as with the sample soln. The method is satisfactory for determining up to 5 mg of **I**. A. O. Jones

4470. **Methods used in industry for the determination of the aromatic hydroxyl function.** R. Goupil and G. Mangeney. *Chim. Anal.*, 1959, **41** (1), 18–22.—In the control laboratory, three methods are used, namely (*i*) *acetylation*, on the macro or micro scale, which determines the hydroxyl group directly, (*ii*) *coupling*, which is an indirect method only, and (*iii*) *esterification*, which determines the hydroxyl group directly, and can to some extent distinguish between aromatic and non-aromatic hydroxyl groups. These methods are outlined, with references and notes. With respect to the analytical laboratory, it is noted that, since it usually deals only rarely with pure chemicals, it is usually necessary to isolate, and identify qualitatively, the constituents possessing aromatic hydroxyl groups, before the analysis proper. The importance of the assistance given by spectrophotometric and allied techniques and chromatographic procedures is briefly noted. (36 references.) R. E. Essery

4471. **Anomalous alkoxyl values for *tert.*-butyl-substituted phenols.** A. D. Campbell and V. J. Chettleburgh (Microanal. Lab., Chem. Dept., Univ. of Otago, Dunedin, New Zealand). *Analyst*, 1959, **84**, 190–193.—The high values obtained when Zeisel's method as modified by Vieböck and Schwappach (*Ber.*, 1930, **63**, 2818) is used on the micro scale for the determination of alkoxyl groups in *tert.*-butyl-substituted phenols can be avoided by replacing the 25% Na acetate soln. used in the scrubber by a mixture of equal vol. of 5% soln. of $CdSO_4$ and $Na_2S_2O_3$. The apparatus is a slight modification of that of Elek (*Ind. Eng. Chem., Anal. Ed.*, 1939, **11**, 174). A 30-ml portion of phenol is added to the sample and then $\simeq$0·2 ml of 30% H_3PO_2 and 1 ml of HI. The liquid is gently refluxed with CO_2 passing at $\simeq$4 ml per min. The scrubber contains 2 ml of the $CdSO_4$ - $Na_2S_2O_3$ soln. and the absorber contains 5 ml of a soln. prepared by adding 10 drops of Br to glacial acetic acid containing K acetate. The contents of the absorber are added to 2 ml of 25% aq. Na acetate soln., Br is destroyed with formic acid, 50 ml of 10% H_2SO_4 is added and the liberated iodine is titrated with 0·02 *N* $Na_2S_2O_3$. A. O. Jones

4472. **Rapid analysis of metol and quinol.** Chien-Teh Soong. *Cinema Tech.*, 1958, (10), 25–26.—The method is based on the fact that metol is insol. in ethyl acetate. The sample containing metol and quinol is shaken with ethyl aectate, then H_2SO_4 or

HCl is added, and the various layers are separated and titrated with $Ce(SO_4)_2$ soln. The procedure is described. The end-point is sharp and the error is <2%. The time required for the determination is only 10 to 11 min. SCI. ABSTR. CHINA

4473. **Quantitative analysis of mono- and poly-basic carboxylic acids in potassium bromide discs by infra-red spectroscopy.** D. E. Nicholson (Humble Oil and Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 519–523.—The method utilises the pressed-disc technique with careful control of mixing time, die pressure and pressing time; the critical variable is mixing time. The method was applied to 8 acids (derived from 1:2:4-trimethylbenzene and the three tetramethyl-benzenes) in admixture and gave an over-all precision of ± 5%. The accuracy was generally better than ± 10% .for normalised compounds. Wavelength data are listed and some anomalies in the spectra are discussed. G. P. COOK

4474. **Determination of the nitrating agent requirements of organic compounds for analytical purposes.** G. Halfter (J. R. Geigy A.-G., Grenzach/Baden, Germany). *Z. anal. Chem.*, 1959, **165** (4), 275–277.—Two methods are described. (*i*) The substance is mixed with H_2SO_4 in a nitrometer, a known excess of HNO_3 is added and the mixture is set aside to allow the nitration to take place. The excess of HNO_3 is then measured by the usual nitrometer procedure, and deducted from a blank conducted in the absence of the test substance. This procedure is applicable to easily nitrated substances. (*ii*) The nitrating acid is added from a burette to the substance in conc. H_2SO_4 maintained under suitable nitrating conditions, the potential being measured by a platinum - calomel electrode pair, and the "Colora-Redoxomaten" (Halfter and Köhler, *Chem.-Ing.-Tech.*, 1958, **31**, 340) is introduced. This measures out the nitrating agent according to the reaction velocity, and shuts off the supply precisely at the end of the nitration. This apparatus not only enables the total requirement to be determined rapidly, but, in the case of substances which are nitrated in stages, enables the portion still unused to be rapidly and certainly determined.
G. P. COOK

4475. **Quantitative determination of the three isomeric chloronitrobenzenes when present together by using infra-red absorption spectra.** V. A. Pozdÿshev and É. S. Levin (K. É. Voroshilov State Scientific-Res. Inst. of Organic Intermediates and Dyestuffs, Moscow). *Zhur. Anal. Khim.*, 1959, **14** (1), 128–132.—The i.r. absorption spectra of *o*-, *m*- and *p*-chloronitrobenzene in CCl_4 differ in having peaks at the following frequencies—*o*-isomer 1053 cm^{-1}; *m*-isomer 1063, 917 and 879 cm^{-1}; *p*-isomer 533 cm^{-1}. Calibration curves are constructed for the percentage (w/v) of the *m*- and *p*-isomers *vs.* percentage transmission at 879 and 533 cm^{-1}, respectively. With a content of *m*-isomer of 0·5 to 3% the error is ≯10% (relative); if the content is >5% the error is about 1%. The absorption of the *m*-isomer at 917 cm^{-1}, though weak, may be used as a qual. test. C. D. KOPKIN

4476. **Spectrophotometric determination of polycyclic aromatic hydrocarbons.** A. J. Lindsey (Sir John Cass Coll., London, England). *Anal. Chim. Acta*, 1959, **20** (2), 175–181.—Previously published methods for the chromatographic separation and spectrophotometric determination of μg quan of polycyclic aromatic hydrocarbons (*cf.* Con *et al.*, *Brit. J. Cancer*, 1954, **8**, 296) are elabo The methods are here applied to samples of c black, soot, solids from air, smoke from to and wood, coal tar and a wide variety of subst that have been treated or contaminated with s The chromatographic elution sequence is give 22 hydrocarbons, together with their u.v. abso characteristics. H. N

4477. **Detection of anthraquinone and anthr in spot-test analysis.** F. Feigl and E. Jur (Lab. da Prod. Mineral, Min. da Agric., R Janeiro, Brazil). *Microchem. J.*, 1959, **3** (2), 214.—*Procedure*—Mix in a micro test-tube micrograms of anthraquinone or a derivative 1 drop of 5 *N* NaOH and several centigran $Na_2S_2O_4$, and warm over a micro flame. A mo less intense red colour appears rapidly. For an cene, allow a drop of benzene or ether soln. co ing 5 to 10 μg of the compound to evapora dryness. Test the residue for anthraquinone. limits of detection are (in μg)—anthraqui 0·5; anthracene, 5·0; 1-aminoanthraquinone, 2-chloro-3-methylanthraquinone, 2·0; an quinone-1- and -2-sulphonic acids, 1·0; and an quinone-1:5- and -2:6-disulphonic acids, 2·0.
D. F. PHILL

4478. **Separation and identification of car genic polynuclear aromatic and related compo by paper chromatography: 9-anthraldehyde, benzanthrone, 3:4-benzopyrene, chrysene, 1:2 dibenzanthracene, 3-methylcholanthrene, phe thraquinone and pyrene.** L. C. Mitchell an Banes (Div. Food, Food and Drug Admin., W ington, D.C., U.S.A.). *J. Ass. Off. Agric. C* 1959, **42** (1), 161–168.—The compounds named be separated on paper impregnated with dime formamide in diethyl ether, development effected with 2:2:4-trimethylpentane. Al atively, but less satisfactorily, soya-bean o diethyl ether is used for impregnation and *iso*propyl alcohol or *n*-propanol for develop The chromatograms are viewed in light of w length 3660 A or 2537 A. The use of a chromo spray of tetrachlorophthalic anhydride in ac improves the identification. A. A. ELDRID

4479. **Diagnostic reactions for the penta triterpenes.** M. Shamma (State Univ., Unive Park, Pa., U.S.A.). *Drug Standards*, 1959, **27** 42–49.—The reactions of the following rea with terpenes of the amyrane, oleanane and lu groups are reviewed—bromine, hydrogen (cata hydrogenation), cold perbenzoic acid, ozone, s ium dioxide in acetic acid, N-bromosuccinimide strong acids. A. R. ROGE

4480. **Detection and determination of pyr and its derivatives.** W. Nielsch (J. A. Benc Co., Ludwigshafen, Germany). *ChemikerZtg*, **82**, 137–138.—The method makes use of the c change (red to violet) when pyridines are tr in a pH 5 buffer with ClCN, formed *in situ* chloramine T and KCN. Nicotinic acid, its d atives and *iso*nicotinic acid (**I**) give colours, carbonyl derivatives of isoniazid fail to give a c until after oxidation with Ce^{2+}. The determin of **I** in the range 0·001 to 0·005 mg is describe
CHEM. ABS

1. **Titanometric determination of the N-oxide in pyridine N-oxide and related compounds.** Brooks and P. D. Sternglanz (Olin Mathieson Corp., New Haven, Conn., U.S.A.). *Anal.*, 1959, **31** (4, Part I), 561–565.—The func- N-oxide group is quant. reduced by $TiCl_3$ a two-electron change, and a 5-min. reaction is adequate for most of the compounds igated. The excess of $TiCl_3$ is determined by on with $(NH_4)_2SO_4.Fe_2(SO_4)_3$ to an orange- nd-point, and a blank determination is run the same conditions as for the sample. al compounds require the presence of a exing agent (citrate or SCN^-) during the tion. The precision and accuracy are within , and the results agree well with those obtained ner methods. G. P. Cook

2. **Determination of sulphur traces in naphthas np combustion and spectrophotometry.** R. W. and J. E. Barney, II [Res. Dept., Standard o. (Indiana), Whiting, U.S.A.]. *Anal. Chem.*, **31** (4, Part I), 596–597.—The method described e used to determine S in concn. from 1 to 400 . After burning the naphtha sample in an M lamp combustion apparatus, the H_2SO_4 d is treated with Ba chloranilate to form anilic acid, which absorbs at 330 mμ and μ. By using a buffer (0·1 *M* in both acetic and Na acetate) the need to adjust the pH is nated. The method is more precise than, at as accurate as and four times as sensitive as idely used method of lamp combustion and dimetry. K. A. Proctor

3. **Potentiometric titration of coal in a non- us medium.** W. J. S. Pringle (Coal Survey Nat. Coal Board, Birmingham, England). *re*, 1959, **183**, 815–816.—Groups containing oxygen are determined in coal with *n*- amine as solvent and Na methoxide in ben- methanol as titrant. The progress of the ion is followed potentiometrically. Sixteen of various ranks and carbon content were sed; those of high carbon content (>90%) ed little or no differentiation and a low acidity, t those of low carbon content (79 to 86%) had her acid content; the curves, however, were ult to interpret. G. P. Cook

4. **Determination of iodine affinity of starch mperometric titration.** Masaharu Ito and kazu Yoshida (Fac. of Agric., Univ. of Agric. Technol., Fuchu, Tokyo). *Bull. Agric. Chem. Japan*, 1959, **23** (1), 34–39 (in English).— h was titrated with iodine, with a rotating ium electrode and a S.C.E. No external was applied and the only instrument necessary a sensitive galvanometer. By plotting free e against added iodine a sharper end-point was ned than by the potentiometric routine of s *et al.* (*J. Amer. Chem. Soc.*, 1943, **65**, 143). disadvantage of the amperometric titration the variability of electrode sensitivity. The od was used to follow the change of iodine- ng capacity of whole and fractionated starches g β-amylolysis. E. Dux

5. **New columnar and mixed-bed ion-exchange ods for surfactant analysis and purification.** . Ginn and C. L. Church (Monsanto Chem. Co., on, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (4, I), 551–555.—The analysis of mixed anionic non-ionic detergents is based on the adsorption of the anionic material on an ion-exchange resin after removal of cations on a strongly acid resin. The non-ionic detergent remains in the percolate and is recovered. The anionic material is eluted from the column with NaOH [2% in *iso*propyl alcohol - water (1:1)] and is finally isolated as a sodium salt. A mixed-bed batch method is also given for the rapid purification and analysis of non-ionic surfactants. The accuracy for both methods is within ± 5%. G. P. Cook

4486. **The equivalent weights of wetting agents.** G. Russell and E. Whittaker (Ramsden Lab., Ilford Ltd., Woodman Rd., Brentwood, Essex, England). *Analyst*, 1959, **84**, 253–254.—A column of Zeo-Karb 225 is converted into its H form by passing 3 *N* HCl through it and then washing it free from acid. The column is then warmed by passing water at 60° through it. A warm aq. soln. of 0·5 g of the wetting agent in 50 ml is applied to the column followed by warm water until 200 ml of eluate has been collected. This is titrated with 0·1 *N* NaOH, with methyl red as indicator. Water (50 ml) is used in place of the sample soln. to determine the blank value of the resin bed. Results with both anionic and cationic wetting agents show satisfactory agreement with those of other methods except with Teepol 530, this exception being due to the presence of potassium salts in the product. A. O. Jones

4487. **Use of phthalic anhydride for determining degree of substitution in hydroxyethylcelluloses.** J. Quinchon. *Compt. Rend.*, 1959, **248** (2), 225–228. —After reaction with a fivefold excess of a 5% (w/v) soln. of phthalic anhydride in pyridine at 40° for ≃5 hr., all the substituent hydroxyl groups in a hydroxyethylcellulose are neutralised. The degree of substitution of the sample can be then determined by back-titrating the unused phthalic anhydride with 0·2 *N* NaOH. The substitution modulus (obtained by an alkoxyl determination) of the sample should be known. For samples having degrees of substitution of ≃0·7, the accuracy is within 6%, but falls to ±12% when the degree of substitution is only ≃0·3. W. J. Baker

4488. **Determination of microgram quantities of mercury in paper.** L. G. Borchardt and B. L. Browning (Inst. Paper Chem., Appleton, Wis.). *TAPPI*, 1958, **41** (11), 669–671.—Mercury (1 to 10 p.p.m.) is determined in 1 g of paper by burning the sample in an oxygen bomb containing 15 ml of 0·1 *N* $KMnO_4$ and 10 ml of 10% H_2SO_4. After ignition of the paper, the bomb is shaken for 2 min., the pressure is released and the contents are transferred to a separating-funnel. Hydroxyammonium sulphate soln. (20%) (5 ml) is added, followed by 5 ml of dithizone soln. (10 p.p.m. in $CHCl_3$). The extinction of the $CHCl_3$ layer is measured against the dithizone soln. at 490 mμ. W. Holden

4489. **Methoxyl groups in wood—Revision of official standard T2m-43.** *TAPPI*, 1958, **41**, 168A–170A.—The new method is adopted from ASTM D914–50 and the apparatus and reagents of T2m-43 are changed. The principle is the same as in the original Zeisel method except that the CH_3I is collected in an acetic acid soln. of K acetate [100 g of anhyd. K acetate in 1 litre of a soln. containing glacial acetic acid (900 ml) and acetic anhydride (100 ml)] containing bromine. The apparatus consists of a reaction flask with side-arm for the admission of CO_2, an air condenser with

trap, and a receiver. The trap is half filled with re phosphorus slurry (0·06 g, in 100 ml of H_2O) and 19 to 20 ml of bromine soln. (5 ml of bromine in 145 ml of K acetate soln.) is placed in the receiver. The weighed specimen, in a gelatin capsule, is heated in the reaction flask on an oil bath at 150° for 40 min. with 2 ml of phenol or, preferably, propionic anhydride, and 6 ml of HI, while CO_2 is passed through. The receiver contents are washed into 10 ml of 22% Na acetate soln., and 90% formic acid soln. is added dropwise until the colour is discharged, followed by 6 drops in excess. After 3 min., 3 g of KI and 15 ml of H_2SO_4 (1:9) are added, and the soln. is titrated in the usual manner with 0·1 *N* $Na_2S_2O_3$. Results from a blank carried out with a gelatin capsule and 2 ml of propionic anhydride are deducted. R. AICHER

4490. **Microscopic identification of man-made fibres from the criminalistics point of view.** A. Longhetti and G. W. Roche (State Crime Lab., St. Paul, Minn.). *J. Forensic Sci.*, 1958, **3**, 303–329.—A scheme for the identification of 18 common synthetic fibres is presented. The scheme requires microscopic examination of longitudinal sections; optical properties such as sign of elongation, birefringence, refractive index; m.p. and eutectic-point with *p*-nitrophenol; solubility and density. Most common synthetics are probably identifiable by this suggested scheme. Single fibres are adequate for the analysis. Difficulties are encountered only with some deeply dyed fibres, particularly black ones. CHEM. ABSTR.

4491. **Determination of low chlorine concentrations in plastics.** D. M. Colman (Radiation Lab., Univ., Livermore, Calif.). *U.S. Atomic Energy Comm.*, Rep. UCRL-5346, 1958, 5 pp.—The method is based on combustion in a Parr oxygen bomb, an ion-exchange step to remove interfering cations, and a mercuric perchlorate titration, with diphenylcarbazide as indicator. The precision is of the order of 0·005%. NUCL. SCI. ABSTR.

4492. **Rapid method for the determination of free alkali in paste rosin sizes.** E. Strazdins. *TAPPI*, 1958, **41** (10), 551–553.—Theoretical considerations are presented which show that the "free alkali" in paste rosin sizes consists predominantly of $NaHCO_3$. A simple apparatus is described in which the "free alkali" is read directly by employing a mercury manometer and liberating the CO_2 equiv. with a mineral acid. *Procedure*—Add *iso*propyl alcohol (5 ml) in small portions to the size sample (15 ± 0·1 g). Stir after each addition until a uniform paste is obtained. Add carefully a soln. (40 ml) of the reagent [*iso*propyl alcohol - H_2SO_4 (sp. gr. 1·84) - H_2O, 100:9:12, by wt.] so that separate layers are obtained. Connect the 125-ml Erlenmeyer flask containing the mixture to a mercury manometer and shake the apparatus until the mercury level is constant. Read the "free alkali" expressed as Na_2CO_3 (%) directly from the scale. The method can be used for all paste rosin sizes, and shows good precision. K. R. C.

4493. **Analysis of rosin size.** E. Strazdins and E. H. Sheers (American Cyanamid Co., Res. Div., Stamford, Conn.). *TAPPI*, 1958, **41** (11), 658–660.—The TAPPI standard method for conventional rosin size is applicable to fortified size except for the determination of free alkali (Strazdins, *Anal. Abstr.*, 1959, **6**, 4492) and unsaponifiables, in which saponification with 2 *N* KOH in place of 0·5 *N* soln. gives increased accuracy and speed. W. HOLDEN

4494. **Identification of pigments in paint pro by infra-red spectroscopy.** T. R. Harkins, Harris and O. D. Shreve (Marshall Lab., E. Pont de Nemours & Co., Inc., Philadelphia, *Anal. Chem.*, 1959, **31** (4, Part I), 541–545.— separation of the pigment from other ingredien is examined by i.r. spectroscopy and classifie organic or inorganic. Its spectrum is then pared with those of reference materials. centrifuging the paint, the isolated pigme washed with acetone - toluene, dried, ground mulled in liquid paraffin preparatory to exar tion. Most inorganic pigments give simple spe a few exhibit no absorption in the infra-red. Sp of organic pigments are much more complex. technique permits the identification of sp compounds in a mixture, and small amoun specific organic pigments can often be identifi the presence of large amounts of inorganic pigm Reference spectra for 21 inorganic and 5 or pigments are given. P. D. PARR-RICHA

4495. **Determination of unsaturation distribu in polybutadienes by infra-red spectrometry.** Silas, J. Yates and V. Thornton (Phillips Petro Co., Bartlesville, Okla., U.S.A.). *Anal. C* 1959, **31** (4, Part I), 529–532.—The absorptic *cis*-1:4 addition in the 12 to 16-μ region studied. The *cis*-1:4 absorption band at 13·5 high *cis*-polymers varies in position and shape a amount of *cis*-1:4 addition changes, and an empi function of the area between 12·0 and 15·75 used to measure it. *Trans*-1:4 and 1:2(v addition are measured at 10·3 and 11·0 μ, res ively. The method was applied to a numb polybutadienes and gave average deviation ⩾ 0·2% for 4 determinations on each polymer by two operators. G. P. Co

4496. **Study of the "Lehigh" or over-all bas of chrome-tanned leather and the determinatio chromic oxide.** Mineral Leather Commi American Leather Chemists Association. *J. A Leath. Chem. Ass.*, 1959, **54** (1), 2–11.—It has shown that the $HClO_4$ oxidation method for d mining Cr_2O_3, which is required for the calcul of the "Lehigh" basicity, gives low results. method is described whereby the error in technique may be checked. *Determinatio "Lehigh" basicity*—To the leather sample 500-ml flask are added 0·2 *N* H_2SO_4 (25 ml), K oxalate soln. (25 ml) and water to 100 ml, the mixture is heated under reflux for 1 hr. cooling, the material is transferred to a 40 beaker, diluted to 200 ml, and titrated with 0 NaOH, with stirring, to pH 7·5 (pH me titrating slowly near the end-point. The bas of the leather may then be calculated by mea the formula given. Two blanks are run.

C. A. SLAT

4497. **Semi-micro determination of acidity chrome leathers.** N. W. von Bassenheim (Copl S.A. Buenos Aires). *Inf. Quím. Anal.*, 1959 (1), 8–13.—The finely divided sample (1·2 g heated with H_2O (25 ml) and 0·1 *N* trieth amine (25 ml) for 45 min. on a water bath, and resulting mixture is cooled and filtered into 0 H_2SO_4 (25 ml). Formaldehyde soln. (40%) (5 is added and the vol. is made up to 250 ml. acidity is determined by titration with 0·1 *N* Na with phenolphthalein as indicator with v leathers or potentiometrically with other sam the results being expressed as H_2SO_4 (%).

l gives results agreeing with those by the l of Riess and Papayannis and has the ad- e that triethanolamine is non-volatile.
G. H. FOXLEY

. **Titrimetric determination of nitrogen in llulose and of nitrocellulose in double-base ents.** R. H. Pierson and E. C. Julian (U.S. Ordnance Test Sta., China Lake, Calif.). *Chem.*, 1959, **31** (4, Part I), 589–592.—The l is based on the reduction of nitrocellulose e^{2+} in acid soln., followed by titration of the so produced with $TiCl_3$, with NH_4SCN as cor. Propellents are first extracted with romethane or acetic acid soln. The over-all on is approx. 0·02% absolute. G. P. COOK

. **Determination of lead styphnate in priming sitions used in explosives.** H. C. J. Saint and wson (Res. Dept., I.C.I. Ltd., Metals Div., h Works, Witton, Birmingham, England). *st*, 1959, **84**, 183–187.—The sample in a 1-ml ain micro-extraction crucible with a porous s extracted in a micro extraction apparatus 0·5-ml additions of aq. 10% ammonium e soln., with alternate water washings, until tract is colourless. The thoroughly washed le is dried at 110° for 30 min., cooled and ed. The ammonium acetate extract is ed to 100 ml, its temp. is adjusted to 20° by sion in a water bath, the absorption cell is its temp. is re-adjusted to 20° and the extinc- f the liquid is measured with Ilford No. 601 (412 mμ) and referred to a calibration prepared with soln. containing 1 mg of lead nate per ml. The temp. of the cell is ob- after the measurement and, if necessary, tinction is corrected to that at 20° by means ormula provided. The method is applicable mples of priming composition weighing ng. A. O. JONES

. **Alkalimetric determination of sodium in ropellents.** G. Norwitz (Pitman-Dunn Lab., ford Arsenal, Philadelphia). *Anal. Chem.*, **31** (4, Part I), 574–578.—Sodium sulphate is as a peptising agent in ball propellents. An ved method for its determination by pptn. as n zinc uranyl acetate (**I**) and titration with I is proposed; the effects of temp. and time e pptn. of the sodium salt, and of soln. and on vol. on the volumetric stage are discussed. *dure*—Heat the sample (10 g) with HNO_3) for 15 min. and evaporate to incipient dry- Add HNO_3 (30 ml) and 70% $HClO_4$ (7 ml) e moist residue in a covered beaker and rate to fumes. Remove the cover and rate to dryness; add H_2O (15 ml), cover and r 3 min. Cool and filter to remove graphite, rate the filtrate to 5 ml, wash down the r with H_2O and evaporate to 3 ml. To the soln. add 20 ml of zinc uranyl acetate soln. t aside for ≮ 1 hr. Filter through a medium ed glass crucible and wash with satd. ethanolic . Dissolve the ppt. in H_2O (50 ml), add lphthalein and titrate with 0·1 *N* NaOH. m a blank determination. The standard ion was ± 0·0024 (11 determinations) for a e containing 0·06% of Na_2SO_4.
P. D. PARR-RICHARD

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also Abstracts—**4254**, Chronopotentiometric nination of mercaptobenzothiazole and other oxidisable org. compounds. **4367**, Determination of S in org. compounds. **4402**, Iodide in photographic soln. **4609**, Anionic detergents in water and sewage. **4619**, Dust-free evaporation of soln. for crystallisation.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

4501. **Application of the microdiffusion technique to biochemical analysis.** P. Tolnay (Biochem. Dept. of Pharm. Res. Inst.). *Magyar Kém. Lapja*, 1958, **13** (10–12), 435–439.—The apparatus for microdiffusion and applications to the determination of the ammonia group, organic acids, halogens, and carbonyl and other compounds are reviewed. (26 references.) G. SZABO

4502. **Rapid determination of calcium in biological material.** H. D. Appleton, M. West, M. Mandel and A. M. Sala (Dept. of Clin. Chem., Met. Hosp., New York, U.S.A.). *Clin. Chem.*, 1959, **5** (1), 36–44. —The serum (0·1 ml) or urine (0·5 ml) is titrated with EDTA soln., with calcein as indicator.
H. F. W. KIRKPATRICK

4503. **Automatic titration of calcium or magnesium in blood serum.** H. V. Malmstadt and T. P. Hadjiioannou (Dept. of Chem., Univ. of Illinois, Urbana, U.S.A.). *Clin. Chem.*, 1959, **5** (1), 50–56.—The use of the Sargent - Malmstadt "Spectro" titrator is described. The titration of Ca is carried out at pH 13 with EDTA, with Calcon (*cf.* Hildebrand and Reilley, *Anal. Abstr.*, 1957, **4**, 2518; Lott and Cheng, *Ibid.*, 1957, **4**, 3576) as indicator; Ca and Mg are titrated together with EDTA at pH 10, with Eriochrome black T as indicator. A single determination takes about 5 min.
H. F. W. KIRKPATRICK

4504. **Determination of magnesium and calcium in urine.** R. Carubelli, W. O. Smith and J. F. Hammarsten (Vet. Admin. Hosp., Oklahoma City, Okla., U.S.A.). *Clin. Chem.*, 1959, **5** (1), 45–49.—Calcium is pptd. from urine as oxalate and the Mg in the supernatant liquid is titrated with EDTA, with Eriochrome black T as indicator. The Ca oxalate is dissolved and the Ca^{2+} are titrated with EDTA, with murexide as indicator.
H. F. W. KIRKPATRICK

4505. **Rapid routine determination of serum magnesium.** H. J. Claes, K. de Doncker and N. Rosselle (R.K. Univ., Louvain, Belgium). *Chem. Weekbl.*, 1959, **55** (4), 39–40.—The sum of Mg and Ca in 0·5 ml of serum can be determined by titration at pH 10·5 with EDTA (disodium salt), with Eriochrome black T as indicator. A photo-electric colorimeter is used for the end-point determination. On a second sample Ca is determined by titration at pH 12·5, with murexide as indicator.
G. BURGER

4506. **Simple procedure for the determination of strontium-89 and strontium-90 in urine and sea water.** J. Kooi (Reactor Centrum Nederland, The Hague). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958,

Rep. A/CONF.15/P/549, 7 pp.—The advantages of the method previously described (*Anal. Abstr.*, 1958, **5**, 3184) are discussed.

4507. **Estimation of strontium in animal bone using X-ray fluorescence analysis.** W. M. B. Roberts (Bur. of Min. Resources, Canberra, Australia). *Nature*, 1959, **183**, 887–888.—Unlike other methods of determining Sr in biological material, this method does not require destruction of the sample. The spectra of the major constituents, Ca and P, do not interfere with the Sr K_α line used and the effect of any heavier elements can be ignored because of their low concn. No internal standard is necessary. A special method of sample preparation designed to ensure absolute reproducibility is described. K. A. PROCTOR

4508. **Determination of thorium in urine.** G. A. Welford, D. C. Sutton, R. S. Morse and S. Tarras (U.S. Atomic Energy Comm., N.Y.). *Amer. Ind. Hyg. Ass. Quart.*, 1958, **19**, 464–468.—Based on the biochemical similarity of Th and Pu, the indicated minimum sensitivity required to study the excretion rate and body burden of Th is 0·2 μg per litre of urine. Several spectrophotometric methods are capable of detecting this quantity of Th. Procedures for using these methods are detailed, and results are given from analyses in which Chrome azurol S (C.I. Mordant Blue 29), thoron and morin were used. NUCL. SCI. ABSTR.

4509. **Determination of iron in horse serum.** I. Ruždić and V. Gregorovič (Cent. Med. Chem. Lab., Zagreb). *Mikrochim. Acta*, 1959, (2), 294–298 (in German).—The method is based on the formation of a coloured complex with 2:2′-dipyridyl reagent (**I**) (6·6045 g of Na acetate is dissolved in 9 ml of glacial acetic acid, 0·5 g of 2:2′-dipyridyl is added and the soln. is diluted to 100 ml). *Procedure*—Serum (2 ml) is mixed with 6 ml of H_2O and 2 ml of aq. trichloroacetic acid soln. (20%) in a test-tube and heated in a water bath at 90° to 95° for 10 min., with occasional shaking. The mixture is then cooled to room temp. and filtered. An aliquot (5 ml) of the filtrate is transferred to a test-tube and 2 ml of **I**, 2 ml of fresh aq. quinol soln. (2%) and 7·5 ml of H_2O are added. A blank determination is carried out at the same time. The extinction is measured at 520 mμ and the concn. of Fe determined by reference to a calibration graph. B. B. BAUMINGER

4510. **Paper-chromatographic determination of mono- and oligo-saccharides in green plants and fruits.** F. Scheffer, E. Welte and K. Müller (Agrik.-chem. u. Bodenkundl. Inst., Univ. Göttingen, Germany). *Z. anal. Chem.*, 1959, **165** (5), 321–337.—Of the three procedures examined, the freezing process has been found advantageous in eliminating changes in the carbohydrate content of green plants and rendering physiological processes inactive. Apparatus for extraction and elution, and paper-chromatographic separation for the qual. and quant. determination of saccharides are described in detail. The limit of error is $\pm$2%. At present the method is limited to saccharides for which reference samples are available. B. B. BAUMINGER

4511. **Determination of aldopentoses in the presence of ketopentoses.** A. Bonsignore, G. Vergano and P. Segni (Ist. Chim. Biol., Univ. Genova, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1958, **34** (7), 324–327.—The method of Dische and Borenfreund (*Anal. Abstr.*, 1957, **4**, 4077) is examined. It is po out that the absorption max. is at 560 mμ in of 552 mμ. L. ZANC

4512. **Chromatography of some phosphoric esters of sugars.** B. N. Stepanenko and A Kuznetsov (Lab. Physiol. Chem., Acad. Moscow). *Biokhimiya*, 1959, **24** (1), 25–32.— chromatography of fructose esters in nine diff acid - organic solvent - water systems shows fructose 6-phosphate (**I**) always migrates rapidly than fructose 1:6-diphosphate Attempts are made to discover why **II** has iously been stated to move as fast as or faster **I** with the same solvents. This can happen i experiment is stopped too soon or if there procedural errors, the most important of whi the use of a non-specific developer. Diph amine is proposed as a developer; this gives a b violet colour with **I** and with fructose, and a colour with **II** and with fructose 1-phosphate. diphenylamine (1 g) is dissolved in conc. H (1·5 ml) and made up to 100 ml with glacial a acid. The dry chromatograms are sprayed the developer and heated for 5 min. at 80° to Some sugar phosphates when used in relat large amounts give two spots of equal colou tensity with some acid solvents. Treatment o paper with HCl reduces the amount of ester b the level at which this effect is produced. K. R.

4513. **Modified system for paper ionophoresis chromatography of phosphate esters and org acids.** V. C. Runeckles and G. Krotkov (Qu Univ., Kingston, Ontario, Canada). *Arch. Bioc Biophys.*, 1959, **80** (1), 94–96.—Improved resolu of the compounds is attained in the method scribed previously (*Anal. Abstr.*, 1958, **5**, 642 electrophoresis in 2·5% pyridine in 0·25% acetic acid by applying d.c. at 800 V (20 V per Chromatography in the second dimension is 2-methoxyethanol - pyridine - glacial acetic a water (8:4:1:1) containing 0·15% (w/v) o hydroxyquinoline. W. H. C. SHA

4514. **Sensitive and rapid method for the d mination of citrates in biological materials.** A Safronov. *Biokhimiya*, 1959, **24** (1), 123–12 Citric acid is oxidised electrolytically to pe bromoacetone, which gives a colour reaction pyridine and alkali. An apparatus is descri *Procedure*—Into the electrolytic cell introduc to 40 μg of citric acid (not exceeding 4 ml), H_2SO_4 (0·5 ml), 8% $MnSO_4$ soln. (0·1 ml), *N* (0·2 ml) and 40% H_3PO_3 soln. (0·25 ml) and r the vol. up to 5 ml. Pass a current of 0·2 for 10 min. at 3 V. Disconnect and add to the 1% H_2O_2 soln. (0·5 ml) and 12% sulphosalicylic soln. (0·2 ml). Remove the liquid from the and wash the cell and its electrode with *isooc* (6 ml), adding the washings to the main bod liquid; shake the mixture for 1 min. and allow settle. Transfer 5 ml of the extract to 30% soln. (2 ml) to which pyridine (4 ml) has added, shake vigorously and then immerse water bath at 80° for 3 to 4 min. A pale-pin red colour indicates the presence of citric The colour is measured photometrically at 530 Results for citric acid in blood and urine are qu The method is satisfactory for the determina of 10 to 400 μg of citric acid. K. R.

. **Biological assay for gibberellic acid with edlings.** R. S. C. Aytoun, A. T. Dunn and L. Seiler (Glaxo Lab. Ltd., Sefton Park, Poges, Bucks., England). *Analyst*, 1959, 6–221.—Rice seedlings (selected from seeds ated under specified conditions at 28° for were placed in dishes, specially designed to in the plants in an upright position with their n the treatment soln. of gibberellic acid in ter, and the randomised dishes were placed olyethylene chamber (humidity >90%) and ted at 29° to 30° for 72 hr. with an illumina- f 6 ft.-candles. Preliminary experiments l that either the growth in 72 hr. or the nce between that and the growth in 24 hr. he best results, and the former was used for ience. The statistical examination of the showed that the method is suitable for the bio-assay of gibberellic acid.

A. O. Jones

. **Determination of "direct" alcohol-precip- bilirubin.** A. Pittera (Ist. Pat. Med. Univ. ania, Italy). *Boll. Soc. Ital. Biol. Sper.*, **34** (14), 710–713.—The "direct" bilirubin is ined at acid pH in aq. soln. (0·5 ml of serum, of dizao reagent and 4 ml of 0·025 *N* HCl); he photometric reading the addition of 1 ml l and 4 ml of 95% ethanol allows the "in- bilirubin to react; the total bilirubin is btained. Then 1 ml of 12% Na_2SO_4 soln. is and, after centrifugation, the ppt. containing irect" precipitable bilirubin is removed, and her spectrophotometric reading is made. hese readings the total, "direct" and "direct" cohol-precipitable bilirubin can be derived.

L. Zanoni

. **Spectroscopy of bacterial chlorophylls sep- by paper and cellulose column chromato-** . I. R. Kaplan and H. Silberman (Sydney Australia). *Arch. Biochem. Biophys.*, 1959, 114–124.—Methods are given for the extrac- nd purification of bacterial pigments on nan 3 MM circular papers developed with e and on cellulose powder columns developed 0% benzene in light petroleum to eliminate noids, then with 0·5% *iso*propyl alcohol in etroleum to elute chlorophylls. All solvents turated with H_2S to prevent oxidation. and u.v. absorption curves of the isolated phylls are given. W. H. C. Shaw

. **Rapid colorimetric method for the deter- on of tocopherol and tocopheryl acetate in** . G. Rindi (Univ., Pavia, Italy). *Int. Z. nforsch.*, 1958, **28**, 225–234.—Total toco- in plasma is determined colorimetrically by dified $FeCl_3$ - 2:2′-dipyridyl reaction after e saponification in the presence of ascorbate vent oxidation of the tocopherol. Free erol is determined by the same colorimetric n on another sample of the plasma. Esteri- ocopherol is calculated by difference. A ion is made for interference by carotene.

Chem. Abstr.

. **Enzymic formation of high levels of phosphoglycerate from 3-phosphoglycerate: n and further metabolism. [Determination -diphosphoglycerate.]** I. Krimsky (Div. of nd Physiol., Publ. Hlth. Res. Inst. of City of ork Inc., U.S.A.). *J. Biol. Chem.*, 1959, **234** 3–231.—2:3-Diphosphoglycerate is measured activating effect on the phosphoglycerate mutase reaction in the conversion of phosphoenol pyruvate into 3-phosphoglycerate, catalysed by enolase and phosphoglycerate mutase. The initial rate of decrease in the absorption at 240 mμ, due to the decrease in phosphoenol pyruvate, is proportional to the concn. of 2:3-diphosphoglycerate, when this is present in limiting amounts.

J. N. Ashley

4520. **Estimation of carbohydrates of the glyco lipids in tissues and body fluids.** J. Brückner (Dept. of Path., Med. Sch., Univ. of Otago, Dunedin, N. Zealand). *Clin. Chim. Acta*, 1959, **4** (2), 149–160.—Glycolipids are extracted from pretreated tissues and hydrolysed with acid. The carbohydrate moiety is then determined with orcinol - H_2SO_4 or with Somogyi's copper reagent.

H. F. W. Kirkpatrick

4521. **Determination of volatile fatty acids in bovine blood by isotope dilution.** J. B. Holter, S. Lakshmanan and J. C. Shaw (Maryland Univ., College Park, U.S.A.). *J. Dairy Sci.*, 1959, **42** (2), 358–362.—The levels of acetate, butyrate and valerate in various samples of cows' blood are determined by an isotope dilution method with the appropriate ^{14}C-labelled compounds. The acids are steam-distilled and titrated after concentration and chromatographic separation. Counting is carried out on the dry residues from the titrations. Most of the volatile fatty acid was present as acetate, with a little butyrate and little or no valerate.

W. H. C. Shaw

4522. **Isolation of salicyluric acid from urine.** D. Hostýnová, P. Kovács, R. Dzúrik and T. R. Niederland (Dep. Chem., Pharm. Fac., Komenský Univ., Bratislava). *Farmácia, Bratislava*, 1959, **28** (5), 145–149.—The method of Quick (*J. Biol. Chem.*, 1935, **101**, 475) was modified and its accuracy increased by controlling the course of the isolation by means of paper chromatography. The chromatograms were developed with *n*-butanol - acetic acid - H_2O (10:1:14) in an atmosphere made alkaline by 0·2% aq. NH_3 on Whatman No. 1 or S. & S. 2043 paper by the descending technique. The separated spots were detected in u.v. light after having been sprayed with a soln. of diazotised *p*-nitroaniline and sulphanilic acid or with 0·2% $FeCl_3$ soln.

J. Zýka

4523. **Estimation of catecholamines in urine.** U. S. von Euler and F. Lishajko (Dept. of Physiol., Karolinska Inst., Stockholm, Sweden). *Acta Physiol. Scand.*, 1959, **45** (2–3), 122–132.—The urine (25 ml) is boiled and filtered, EDTA (disodium salt) (0·5 g) is added, and the pH is adjusted to 8·2 to 8·3 with *N* NaOH. The treated urine is passed through aluminium oxide (1 g) in a 10-mm-diam. column at a rate of 1 to 2 ml per min., the column is washed with water (10 to 15 ml) at the same rate, and the catecholamines are eluted with 0·25 *N* acetic acid (2 × 5 ml). The eluate is neutralised to pH 6·2 to 6·3 with *N* aq. NH_3 and a suitable aliquot is oxidised with 0·25% $K_3Fe(CN)_6$ soln. (0·1 ml). After 2 min. a fresh mixture of 20% NaOH soln. and 2% ascorbic acid soln. (9:1) (1·0 ml) is added and the volume is made up to 10 ml with water. (If more than 1 ml of eluate is taken an equal volume of alkali - ascorbic acid soln. is added.) A blank is prepared by adding all the reagents except $K_3Fe(CN)_6$ to an aliquot of the eluate. The fluorescence of sample and blank is measured within 10 min. in a fluorimeter with two sets of filters—(*a*) primary 395-mμ interference filter and secondary

Ilford 623 (490 mμ), (*b*) primary 436-mμ interference filter and secondary Corning 3486 (540 mμ). The results are calculated with the aid of the following formulae, where m and n are the observed fluorescence values (sample — blank) with filter sets *a* and *b*, respectively, and A_a, A_b, N_a and N_b represent the fluorescence per μg of adrenaline and noradrenaline, respectively, determined at the same sensitivity as for the samples—noradrenaline $= y = [(mA_b/A_a) - n]/(N_aA_b/A_a - N_b)$, adrenaline $= (n - yN_b)/A_b$. A lucite rod can be used as a standard after the determination of its fluorescence in terms of adrenaline and noradrenaline. Recoveries of added catecholamines are 70 to 100%.

R. A. BRENAN

4524. **Study of the reaction of adrenaline with ethylenediamine.** G. Nadeau and L.-P. Joly (Dept. of Chem., Univ. Laval, Quebec, Canada). *Canad. J. Biochem. Physiol.*, 1959, **37** (2), 231–244 (in French).—Optimum conditions for the reaction are defined. The necessity for a preliminary oxidation to adrenochrome is confirmed. The reaction pH should be 10·4 ± 0·1 and the extraction of the fluorescent compound is best made with *iso*butyl alcohol. The reacting mixture should contain 114·5 millimoles of ethylenediamine base per 100 ml, and the reaction allowed to proceed for 20 to 30 min. at 50°. Exposure to diffuse or u.v. light affects the yield of fluorescent compound. The beneficial effect of various ions is discussed.

H. F. W. KIRKPATRICK

4525. **Determination of adrenaline and noradrenaline. I. Modified reaction with ethylenediamine.** G. Nadeau and G. Sobolewski (Hôp. St.-Michel Archange, Quebec, Canada). *Canad. J. Biochem. Physiol.*, 1959, **37** (3), 441–451 (in French).—Treatment with molybdate before the addition of ethylenediamine gives reproducible and enhanced fluorescence. The reaction is carried out at room temp. and at 50°, the difference in the two fluorescence readings being a measure of the noradrenaline, and the room-temp. reading may be corrected to give the adrenaline concn. *Procedure*—To the soln. of amines (10 ml) add 1% aq. ammonium molybdate soln. (0·8 ml), and set aside at room temp. in the dark for 30 min. Add a reagent (0·5 ml) composed of ethylenediamine (60 ml) and azeotropic HCl (30·8 ml), both freshly distilled, diluted to 100 ml with H_2O. Set aside for 30 min. as before, extract with *iso*butyl alcohol (10 ml) and read the fluorescence (F_1) of the organic phase. Return the alcohol to the reaction mixture, heat at 50° for 30 min., cool, shake and again read the fluorescence of the alcohol layer (F_2). Correct F_1 and F_2 by deducting the blank fluorescence. Then adrenaline (μg) $= K_1(F_1 - K_2F_2)$, and noradrenaline (μg) $= K_3(F_2 - F_1)$, where K_1, K_2 and K_3 are constants for the apparatus evaluated with known amounts of the amines.

H. F. W. KIRKPATRICK

4526. **Identification of thyroxamine in thyroid and plasma.** G. Hillmann, B. Keil and P. Taslimi (Chem. Lab., Med. Univ.-klinik, Tübingen, Germany). *Z. Naturforsch.*, 1958, **13b**, 820–821.—Butanol extraction of ^{131}I-labelled rat-thyroid homogenates (not hydrolysed) followed by isotopic co-crystallisation with thyroxamine (pptn. from HCl - ethanol soln. with conc. aq. NH_3) separates the thyroxamine from other iodine compounds. Paper chromatography is made with 50% piperidine. Further identification can be afforded by conversion into thyroxamine N-succinate, followed by paper chromatography in *iso*amyl alcohol - 5 *N* aq. NH_3 as well as *tert.*-amyl alcohol - 5 *N* aq. The compound is detected by autoradiography

H. F. W. KIRKPATRI

4527. **Determination of urea by diacetyl oxime.** R. Sardou (Hôpital Purpan, Toul France). *Ann. Biol. Clin.*, 1958, **16**, 53–60.—in either whole blood or serum was determined filtrate, deproteinised with alkaline $ZnSO_4$ treating the filtrate with diacetyl monoxime i presence of a soln. of arsenic acid in H_2SO yellow colour (475 mμ) developed after heatin 5 min. A standard curve was necessary be of the non-linearity of the curve, but results accurate to within ±1% regardless of the ty blood soln. or clotting agent (oxalate or flu used.

CHEM. ABS

4528. **Applications of isatin in the paper chron graphy of amino acids.** U. Bonomi and R. Vecc (Ist. di Patolog. Gen. dell'Univ. di Siena). *Soc. Ital. Biol. Sper.*, 1959, **35** (5), 210–211.—I sprayed on paper chromatograms as a 0·4% in acetone, was used to locate and identify pr hydroxyproline, phenylalanine, histidine, as gine, aspartic acid, cysteine, glycine, cysteic glutamic acid, methionine, norvaline and tr phan. A minimum of ≃2 μg of any amino was detectable by the spray. Individual a acids yielded characteristic colours, which deve on paper within 5 to 24 hr., but (except with p and phenylalanine) not when the reactants mixed in solution.

C. RAINB

4529. **Technique of separation of amino aci ion-exchange columns.** J. Schormüller, Belitz and G. Adler (Inst. f. Lebensmittelche Lebensmitteltechnol., Berlin). *Z. Leben Untersuch.*, 1959, **109** (2), 129–134.—The proc is based, with modifications, on the Moore and technique. A soln. (1 to 2 ml at pH 2·5) c acids (5 to 15 mg) is deposited on a suitably pared column (125 cm high) of Dowex 50-X8 which has previously been washed with a c buffer at pH 3·1. The first elution is carrie with this buffer at 37·5°, and the second w citrate buffer at pH 4·25 at temp. gradually to 60°. A succession of the acid and neutral a acids is thus eluted in 1- or 2-ml portions separate the basic acids, a separate soln. (at p is added to a 20-cm column (previously washe a citrate buffer at pH 5·0), the acid and n amino acids are washed out with a citrate at pH 5·0, after which the basic acids are pr sively eluted at room temp. with a phosphate at pH 6·9 followed by a citrate buffer at p The individual acids in the eluates are deter spectrophotometrically with the use of the and Stein (modified) ninhydrin reagent. Th of a buffer at pH 3·1 on the first column imp the separation of glycine and alanine. The lapping of proline and glutamic acid in respective eluates can be resolved by carryin the optical determination with two different filters. The peak positions for the basic acids slightly from those given by Moore and Stein. max. error of the individual determinati ⩾7%.

P. S. A

4530. **Determination of amino acids in k The content of alanine, glycine, serine and thr in horn.** J. Mareček (Res. Inst. Fats and Prague). *Chem. Listy*, 1959, **53** (2), 194– Hydrolyse the sample (100 g) by boiling wit

(300 ml) under reflux for 24 hr., evaporate rup, dilute with H_2O, filter and wash the . Dilute the combined filtrate and washings ml. The determination of amino acids can ied out (*i*), by measuring the transmittance h photometer) of a methanolic soln. of the ed spots eluted from chromatograms after development by one-dimensional chromato- on Whatman No. 1 paper with butanol - acid - water (4:1:5) and comparison with a tion curve, (*ii*), by the method of descending rd and ascending sample or (*iii*) by the n method of Virtanen *et al.* (*Hoppe-Seyl. Z.*, **193**, 268) for the determination of acet- de formed from alanine by reaction with rin. Satisfactory agreement was found n the three methods. J. ZÝKA

. **Rapid quantitative determination of argi- histidine and lysine by ion-exchange paper atography.** H. R. Roberts and M. G. Kolor Dairy Products Corp., Oakdale, N.Y., U.S.A.). *Chem.*, 1959, **31** (4, Part I), 565–566.—The tive technique of Tuckerman (*cf. Anal. Abstr.*, **5**, 2338) was made quantitative. A 4-hr. ding development with an acetate buffer 2) resolves these compounds as round compact the maximum densities of which are linearly l to the log. of the concn. The standard ion was $< \pm 1{\cdot}5\%$. G. P. COOK

2. **Microchemical determination of histidine tein hydrolysates and urine.** K. Voigt (Inst. ed. and Biol., Acad. of Nat. Sci., Berlin). *m. Z.*, 1959, **331** (2), 127–132.—The method ped is a modification of that of Kapeller - (*Biochem. Z.*, 1933, **264**, 131; 1934, **271**, 206), ch histidine is treated with bromine in acetic oln. and, on the addition of an aq. NH_3 - nium acetate or an aq. NH_3 - ammonium de buffer, a blue or blue - violet colour, tively, is produced. These colours, which Beer's law, are measured photometrically. lue colour is the more sensitive to changes in ine concn., and 0·2 to 0·7 mg of histidine can termined. Cyclic amino acids, methionine, lacetic acid, uric acid, phenol, heavy-metal and excess of bromine affect the intensity and y of the colour produced. In the application method to protein hydrolysates and urine, ities are first removed by treatment with followed by passage of the soln. through ns of activated charcoal and Wofatit C. rine it is necessary to pre-treat with Pb acetate, passage of the H_2S. Results are given for the ine content of normal urine (29 to 65 mg per), and of urine from malignant tumour cases 63 mg per 24 hr.), also for serum albumin n), serum globulin (rat), and hydrolysates experimental tumours in mice and rats. D. B. PALMER

3. **New method of determination of mercapto s in biological preparations.** A. Mussini (Ist. col., Univ. of Milano, Italy). *Boll. Soc. Ital. Sper.*, 1958, **34** (21), 1419–1421.—*Procedure*— mple ($\equiv$ 1 to 4 μg of –SH) is triturated with a re of EDTA and sulphosalicylic acid (1·25 0·335% EDTA soln. plus 1·25 ml of 2·5% osalicylic acid soln.). The mixture is centri- and to an aliquot of the supernatant liquid ml to 1 ml) is added 2 ml of *p*-chloromercuri- ic acid soln. (0·001%), with shaking, followed ml of 0·3 *N* acetic acid and 1 ml of dithizone reagent (100 mg of dithizone in 100 ml of $CHCl_3$; 3 ml of this soln. is diluted to 90 ml with $CHCl_3$). After shaking, the $CHCl_3$ layer is made up to 10 ml and the extinction is read at 620 mμ. L. ZANONI

4534. **Colorimetric determination of cysteine.** H. F. Liddell and B. Saville (Chem. Defence Exp. Estab., Porton Down, Salisbury, Wilts., England). *Analyst*, 1959, **84**, 188–190.—To prepare a calibration graph, soln. of cysteine hydrochloride (L or DL form) containing 1 to 70 μg per ml are made. To a 1-ml portion of each of these are added 5 ml of a soln. of HNO_3 (5 ml of 0·01 *M* aq. $NaNO_2$ added to 40 ml of H_2SO_4 and diluted to 100 ml) and, after 5 min., 1 ml of a 0·5% soln. of ammonium sulphamate and then $\simeq$ 10 ml of a soln. prepared by mixing 4 vol. of a 3·44% soln. of sulphanilamide in 0·4 *N* HCl with 1 vol. of a 1% soln. of $HgCl_2$ in 0·4 *N* HCl. Finally the liquid is diluted to 25 ml with a 0·1% soln. of N-1-naphthylethylenediamine dihydrochloride in 0·4 *N* HCl and the extinction is measured absorptiometrically with Ilford No. 605 filters. The sample soln. is treated similarly. A simplified procedure is described in which the last two soln. are added as a mixture, the loss of accuracy being slight. The reaction is specific for mercapto groups. There is no interference from Zn, but I^- and extremely large amounts of Hg, Ag and Cu^{2+} interfere. A. O. JONES

4535. **Estimation of cysteine and cystine at the mercury-pool electrode.** R. C. Kapoor (Univ. Allahabad, India). *Z. anal. Chem.*, 1959, **166** (1), 1–4 (in English).—The amperometric titration of cysteine and cystine in concn. of 10^{-5} *M* with phenylmercury chloride and with $HgCl_2$ was studied with a mercury-pool electrode. The titrations were conducted under nitrogen in soln. buffered to pH 5 and pH 9·2 in the presence of KCl (0·1 *M* and 2·0 *M*) and, for cystine, Na_2SO_3 (0·01 *M*). Polarograms were recorded manually and automatically. G. BURGER

4536. **Determination of β-lipoproteins of serum with calcium chloride and heparin.** M. Greppi, F. M. Antonini and C. Salvini (Ist. Chim. Med. Gen., Univ., Firenze, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1958, **34** (20), 1339–1343.—The method described is based on the measurement of the turbidity formed by serum in the presence of calcium chloride on the addition of heparin. *Procedure*—Serum (1 ml) containing 10% of $CaCl_2$ is diluted with 10 ml of heparin soln. (1%). The reading is carried out at 700 mμ 20 min. after the addition of heparin. The mean error is $\pm$ 0·67%. L. ZANONI

4537. **New method for photometric determination of small amounts of protein.** W. Heinzel and V. Neuhoff (Path. Inst. d. Univ., Tübingen, Germany). *Naturwissenschaften*, 1959, **46** (4), 146.—The test soln. is spotted on to poly(vinyl chloride) paper ("Rhovyl," S. & S.) previously impregnated with oleic acid, dried and stained with Amido black 10B (C.I. Acid Black 1), as in electrophoresis. The paper is transferred to a glass cell containing just sufficient nitrobenzene to render it transparent, and the extinction is read at 610 mμ in a specially adapted spectrophotometer. H. F. W. KIRKPATRICK

4538. **Sources of error in the paper-electrophoretic determination of serum glycoproteins.** W. Boguth and H. Schnappauf (Vet.-Physiol. Inst. d. Justus Liebig Univ., Giessen, Germany). *Naturwissenschaften*, 1959, **46** (4), 145–146.—Two main sources

of error are noted. Albumin is lost by elution into reagents owing to insufficient fixing, and an error in scanning is introduced by the use of a transparency oil of unsuitable refractive index. To eliminate these errors, the drying temp. of the strip should be 150°, and the refractive index of the oil used for rendering it transparent should be 1·55.
H. F. W. KIRKPATRICK

4539. **Albumin trail in paper protein electrophoresis.** W. B. Yeoman (Frenchay Hosp., Bristol, England). *Clin. Chim. Acta*, 1959, **4** (2), 246–251.—The trail of albumin on Whatman No. 1 paper is of the order of 9 to 14% if 0·01 ml of serum is used. The correction may be conveniently assessed in electropherograms by drawing a line parallel to the abscissa of scan at a height above the abscissa equal to one-third of the height of the minimal trough shown by the scan. All of the area between this line and the abscissa is considered as albumin.
H. F. W. KIRKPATRICK

4540. **Staining properties of Oil Red O and a method of partial purification of the commercial product.** H. Kutt and T. T. Tsaltas (Neurol. Service of the Cornell Med. Div., Bellevue Hosp., New York, U.S.A.). *Clin. Chem.*, 1959, **5** (2), 149–160.—A yellow - brown constituent of commercial Oil Red O (C.I. Solvent Red 27) stains serum proteins so that lipid electropherograms consist of a red lipid pattern superimposed on a brownish protein pattern. Successive treatments of the dye with 60% ethanol by refluxing, cooling and decantation yield a residue consisting principally of a red constituent that moves slowly on paper chromatograms developed with 95% ethanol. This partial purification yields a dye that stains lipoproteins satisfactorily.
H. F. W. KIRKPATRICK

4541. **Chromatography of proteins. III. Human, horse and dog haemoglobins on cation-exchange cellulose.** F. J. Gulter, E. A. Peterson and H. A. Sober (Nat. Cancer Inst., U.S. Dept. Health, Bethesda, Md., U.S.A.). *Arch. Biochem. Biophys.*, 1959, **80** (2), 353–366.—Chromatography of haemoglobins in the carboxy form is carried out on carboxymethylcellulose eluted with 0·01 *M* sodium phosphate buffers to give a gradient of increasing pH. Eluted fractions are examined spectroscopically, and, after concentration, by paper electrophoresis and sedimentation methods. The heterogeneous nature of adult human and horse carboxyhaemoglobins and the effects of temp. and of conc. urea soln. on the chromatography are discussed.
W. H. C. SHAW

4542. **Ferrous iron - sulphuric acid reagent for the determination of pure oestrogens.** E. Epstein, W. O. Maddock and A. J. Boyle (Dept. of Chem. and Med., Wayne State Univ., Detroit, Mich., U.S.A.). *Talanta*, 1959, **2** (2), 183–186.—For the determination of oestrone (**I**) and oestradiol (**II**), measured amounts of soln. are evaporated on a water bath in round Coleman spectrophotometer cells (19 mm × 150 mm). The reagent [290 mg of $(NH_4)_2SO_4.FeSO_4.6H_2O$ (**III**) in 100 ml of 52% H_2SO_4; stable for 3 days if protected from air] (8 ml) is added, the mixtures are heated in a boiling-water bath (50 min. for **I**, or 30 min. for **II**), then cooled and read at 505 mμ. For the determination of oestriol (**IV**), the soln. is evaporated as described above, then treated with 8 ml of reagent (290 mg of **III** dissolved in 7·5 ml of water and made up to 100 ml at room temp. with 70% H_2SO_4), heated as described above for 50 min., then cooled and read at 500 mμ. Results are referred to the appropriate standard curves, which are linear up to 17 (for **I** and **II**) or 33 μg (for **IV**). The reproduci ranges are from 3 to 9% (for **I**), 5 to 8% (fo and 1 to 5% (for **IV**).
R. E. ESSE

4543. **Two-dimensional chromatography, ele phoresis and electrochromatography of ketoste** P. Bianchini and M. Mantovani (Lab. Ric. C Ind. Farmacol., Milano, Italy). *Boll. Soc. Biol. Sper.*, 1958, **34** (18), 1106–1110.—In the dimensional chromatographic method descr butanol - methanol - benzene - H_2O (1:1:1: used as developer. The electrophoresis is ca out with a borate buffer at pH 8·6 at 150 to for 7 to 20 hr. The detection is made with K_2 aniline phthalate or hexamethylenediamine. combination of these methods, chromatograp carried out on paper (30 cm × 30 cm) with starting point on the middle line of the p after development and drying, the chromatogr submitted to electrophoresis after folding it the middle line. With this technique exce separation of ketosteroids can be attained.
L. ZANC

4544. **Estimation of corticosteroids by the r tion of ferricyanide.** N. R. Stephenson (Lab. o Food and Drug Directorate, Dept. of Nat. H and Welfare, Ottawa, Canada). *Canad. J. Bioc Physiol.*, 1959, **37** (3), 391–398.—The α-ke side-chains of the adrenal corticosteroids susceptible to oxidation, and in this metho ferrocyanide produced by the reduction of alk ferricyanide is measured photometrically as Pru blue. Dissolve the steroid in aldehyde-free ethanol and add 0·5 ml of the soln. to $K_3Fe(CN)_6$ soln. (0·5 ml) in a 10-ml glass stopp graduated cylinder. Add 0·23% Na_2CO_3 (1·0 ml), stopper the cylinder and immerse it depth of approx. 4 cm in a 60° constant-t water bath for exactly 60 min. Cool the cyli in water at 15° for 1 to 2 min., add $(NH_4)_2SO_4.Fe_2(SO_4)_3.12H_2O$ soln. containing of 85% H_3PO_4 (1·0 ml) and mix by gentle swi Allow the colour to develop for 15 min. and dilute the reaction mixture with water to 6· 10·0 ml according to the amount of steroid pre Measure the extinction of test and reagent b (0·5 ml of aldehyde-free 95% ethanol treate described above) at 620 mμ. Beer's law is ob over the range 0·005 to 0·050 mg of steroid. relative reducing activities of several corticoste are compared.
R. A. BRENA

4545. **Determination of the activity of st hydrolysing enzymes.** M. Richter (Inst. Ernähr Potsdam-Rehbrücke, Berlin, Germany). *Err ungsforschung*, 1959, **4** (1), 19–34.—Three met depending on (*i*) the change in viscosity, (*ii*) iodine colour, and (*iii*) the reducing power, reviewed. Newer views on the kinetics of enz breakdown are discussed. (49 references.)
S.C.I. ABST

4546. **Simple method for the assay of ca hydrases.** R. W. P. Master (Haffkine Inst., P Bombay, India). *Experientia*, 1959, **15** (3), 123 (in English).—Carbohydrate soln. (10%) (1 m mixed with a dialysed enzyme preparation (2 and buffer soln. of the appropriate pH. The ture is incubated at 37° for 24 hr. Aliquots (1 of the reaction mixture, together with stan sugar soln., are applied to points on the circ ference of a circle (3·0 cm diam.) drawn at

of a filter-paper disc. By using appropriate tographic solvents and chromogenic locating s, the products of the enzyme reaction are termined. The method, which gives reason- curate results when quick determinations are d, is most convenient when the products of ction have R_F values very different from f the substrates. P. NICHOLLS

Electrophoretic behaviour of serum amylase. ination of amylase.] R. L. McGeachin and ewis (Dept. of Biochem., Univ. of Louisville Med., Ky., U.S.A.). *J. Biol. Chem.*, 1959, , 795–798.—The amyloclastic amylase deter- on of Van Loon *et al.* (*Amer. J. Clin. Path.*, 32, 1134) has been modified for use on the cale. The quantities of samples and reagents uced to 10% of the original amounts and ne buffer, pH 7·6, is used instead of phos- buffer, pH 7·0, in some comparative deter- ons. The extinctions of the blue starch - mixtures are determined spectrophoto- lly at 660 mμ. J. N. ASHLEY

New method of demonstrating esterases in G. K. Steigleder and H. Elschner (Univ.- inik, Frankfurt am Main, Germany). *Klin. hr.*, 1959, **37** (2), 104–105.—A reagent that monstrate esterases on the surface of living nd in sections by the production of a grey - olour which changes to black consists of (w/v) 1-naphthyl acetate and 0·1% (w/v) ue salt BB (C.I. Azoic Diazo Component 20), issolved in a few drops of acetone, in 0·1 *M* ate buffer. In the control soln. 1-naphthol s the 1-naphthyl acetate. For living skin, n. is placed in a small glass cell affixed to ected site with adhesive tape. The reaction plete after 25 min. D. B. PALMER

Spectrophotometric method for determina- diamine oxidase (DAO) activity. B. Holm- nd R. Tham (Dept. of Pharmacol., Karolinska Stockholm 60, Sweden). *Acta Physiol.* 1959, **45** (2–3), 152–163.—Diamino com- are oxidised by diamine oxidase to the onding aminoaldehydes. If butane-1:4- e dihydrochloride (putrescine) is used as the ate, the aldehyde formed condenses to the compound $\triangle^1$-pyrroline, and this will react -aminobenzaldehyde to give a yellow com- probably 1:2-dihydro-2:3-trimethylene- olinium hydroxide. The enzyme preparation, sphate buffer *M*/15, pH 6·8, is added to *M* *o*-aminobenzaldehyde, also in phosphate (2·5 ml) and the volume is made up to 4·5 ml he buffer. After temperature equilibration, putrescine in phosphate buffer (0·5 ml) is and the mixture is incubated at 37°, usually r. The reaction is stopped by the addition % trichloroacetic acid (1·0 ml), and after ugation the extinction of the supernatant is measured at 430 mμ. A blank is prepared rying out this procedure, but omitting the n of putrescine. R. A. BRENAN

Purification and properties of glutathione lase of erythrocytes. [Determination of ione peroxidase activity.] G. C. Mills (Dept. ochem., Univ. of Texas Med. Branch, ton, U.S.A.). *J. Biol. Chem.*, 1959, **234** (3), 6.—Two methods of assay are described. first method the ability of the peroxidase- d glutathione system to prevent the oxidative breakdown of haemoglobin is used as a measure of the enzymic activity. The choleglobin formed from haemoglobin is determined by a standard method. In the second method the rate of oxidation of reduced glutathione by H_2O_2 is used as a measure of the peroxidase activity. The peroxidase catalyses the reduction of H_2O_2, with reduced glutathione serving as H donor. Reduced glutathione, which is left at the end of the reaction, is determined by the mercuribenzoate method of Boyer (*J. Amer. Chem. Soc.*, 1954, **76**, 4331). J. N. ASHLEY

4551. **Assay of arylsulphatases A and B in human urine.** H. Baum, K. S. Dodgson and B. Spencer (Dept. of Biochem., Univ. of Wales, Cardiff). *Clin. Chim. Acta*, 1959, **4** (3), 453–455.—The determination of these enzymes independently in the presence of each other is possible from the following observations. Normal kinetics are shown by A at low substrate concn. in the presence of $2{\cdot}5 \times 10^{-4}$ *M* $Na_4P_2O_7$, whilst considerable inhibition of B occurs under these conditions. B is specifically inhibited by Cl^- when potassium 2-hydroxy-5-nitrophenyl sulphate is the substrate, whereas A is not. The determination of B in the presence of A is based on the observation of only a small residual activity after the first 20 min. of reaction on incubation at high substrate concn. in the presence of Ba^{2+} and in the absence of interfering ions. Practical details are given.
H. F. W. KIRKPATRICK

4552. **Simple micro-method for the determination of uropepsin in urine.** R. Ménaché (Lab. Clin. de l'Hôpital "Hasharon," Petah-Tikva, Israel). *Bull. Soc. Chim. Biol.*, 1959, **41** (1), 175–180.—The tyrosine produced by the incubation of 0·2 ml of urine with a haemoglobin substrate for 1 hr. at 37° and pH 1·5 to 2·5 is measured colorimetrically by its reaction with Folin and Ciocalteu's phenol reagent. Tyrosine standards are used for comparison, and the results are expressed as the tyrosine (mg) which would be produced by the 24-hr. urine output. Normal values (for Israel) are taken as 300 to 600 mg of tyrosine per 24 hr.; 3 determinations on the same urine gave values of 440, 470 and 430 mg per 24 hr.
D. W. Moss

4553. **Simple colorimetric method for estimating serum pseudocholinesterase.** R. L. Smith, H. Loewenthal, H. Lehmann and E. Ryan (Chase Farm Hosp., Enfield, Middx., England). *Clin. Chim. Acta*, 1959, **4** (3), 384–390.—The serum is incubated with a buffered phenyl benzoate substrate (pH 8·6) and the liberated phenol is determined with Folin and Ciocalteu's reagent.
H. F. W. KIRKPATRICK

4554. **[Determination of] the distribution of glutamic - γ-aminobutyric transaminase in the nervous system of the rhesus monkey.** R. A. Salvador and R. W. Albers (Lab. of Neurochem., Nat. Inst. of Neurol. Diseases and Blindness, Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1959, **234** (4), 922–925.—The transaminase activity is determined by fluorimetric measurement of the condensation product of succinaldehydic acid (produced from Na γ-aminobutyrate during incubation with the enzyme) with 3:5-dinitrobenzoic acid. Conditions are described which allow measurement of activity on 3 to 15 μg of lyophilised brain tissue. J. N. ASHLEY

4555. **[Determination of] a bacterial pterin deaminase.** B. Levenberg and O. Hayaishi (Nat. Inst. of Arthritis and Metabol. Diseases, Bethesda,

Md., U.S.A.). *J. Biol. Chem.*, 1959, **234** (4), 955–961.—A spectrophotometric method is described, based on shifts in the u.v. spectrum of 2-amino-4-hydroxypteridine-6-carboxylic acid which accompany the conversion of the 2-amino group into the 2-hydroxy group by the deaminase at pH 6·3. The extinction, which is determined at 360 mμ for crude enzyme prep. and at 290 mμ for purer prep., is linearly related to the activity of the enzyme.

J. N. Ashley

4556. **Nephelometric determination of elastase activity and method for elastoproteolytic measurements.** I. Banga, J. Baló and M. Horváth (First Dept. of Path. Anat. and Exp. Cancer Res., Med. Univ., Budapest, Hungary). *Biochem. J.*, 1959, **71** (3), 544–551.—Methods for the determination of elastase activity are reviewed. A rapid and, within certain limits, sufficiently accurate nephelometric method is described in which a photometer is used to measure the changes in extinction of a suspension of elastin granules in the presence of elastase. The extinction is read with a red RG_1 colour filter with max. transmission at 620 to 750 mμ, and the amount of enzyme is ascertained from a standard graph.

J. N. Ashley

4557. **Pyridine nucleotide transhydrogenase. VII. Determination of the reactions with coenzyme analogues in mammalian tissues.** [**Assay of transhydrogenase activity.**] A. M. Stein, N. O. Kaplan and M. M. Ciotti (Graduate Dept. of Biochem., Brandeis Univ., Waltham, Mass., U.S.A.). *J. Biol. Chem.*, 1959, **234** (4), 979–986.—The method, which involves the transfer of hydrogen or electrons from reduced triphosphopyridine nucleotide to the acetylpyridine analogue of diphosphopyridine nucleotide, gives values for transhydrogenase activity much higher than those obtained by other methods. The reaction is carried out at pH 6·5 and 25°, and the activity of the enzyme is determined from the amount of the acetylpyridine analogue of reduced diphosphopyridine nucleotide; this is measured spectrophotometrically at 365 mμ.

J. N. Ashley

4558. **Assay of pepsin using ribonuclease as substrate.** A. Berger, H. Neumann and M. Sela (Weizmann Inst. Sci., Rehovoth, Israel). *Biochim. Biophys. Acta*, 1959, **33** (1), 249–251.—In the method described, a soln. of the sample pepsin is allowed to react at pH 1·7 with ribonuclease for 30 min. at 25°. The reaction is then stopped by the addition of acetate buffer at pH 7, and the residual ribonuclease is determined by the addition of ribonucleic acid, adjustment to pH 5 and incubation at 25° for 25 min. Uranyl acetate in 25% $HClO_4$ is added and the extinction of the supernatant liquid obtained by centrifugation is measured at 260 mμ. The graph of extinction *vs.* concn. of pepsin is rectilinear up to 15 μg of sample.

W. H. C. Shaw

4559. **Paper-chromatographic assay for ribonucleases using cyclic mononucleotides as substrates.** J. T. Nodes (Chester Beatty Res. Inst., Royal Cancer Hosp., London, England). *Biochim. Biophys. Acta*, 1959, **32** (2), 551–553.—In the method described, the sample (0·1 ml) is added to 0·1 *M* acetate buffer at pH 6·0 (0·1 ml) with 0·1 ml of a solution of adenosine (or cytidine) 2′:3′-cyclic phosphate (15 mg per ml) as substrate. After incubation at 37° for 45 min. or 120 min. for the respective substrates, the reaction is stopped with 0·6 ml of ethanol - diethyl ether - $CHCl_3$ (2:2:1). After evaporation to about 0·4 ml during 10 min. at 85°, portions of the supernatant liqui applied to Whatman No. 1 paper and develop the descending technique for 16 hr. with $(NH_4)_2SO_4$ soln. - *iso*propyl alcohol - *M* Na a (40:1:10). Areas corresponding to the una 2′:3′-cyclic phosphate and to the 2′- and 3′ phates are cut out and eluted with 5 ml of and the extinctions are measured at 2 (adenosine) or 270 mμ (cytidine). The prop of substrate hydrolysed is then calculated fro extinctions.

W. H. C. Sh

See also Abstracts—**4276**, Determination of Na in tissues. **4291**, Determination of Be in and bone. **4396**, Determination of F in **4446**, Determination of acetyl groups in ste **4459**, Separation of isomaltose and gentio **4562**, Separation of alkaloids from urine **4567**, Determination of flavonoids in tol **4569**, Determination of curcumin. **4641**, Ele for intra-vascular O determination. **4652**, S of error in counting ^{14}C.

Pharmaceutical analysis

4560. **Determination of water in drugs cont volatile oils by means of the Karl Fischer m** M. Šaršúnová, Ľ. Čičmancová and J. Menk (Control Pharm. Lab., Bratislava, Czechoslov *Farmácia, Bratislava*, 1959, **28** (2), 55–57.— Karl Fischer method is recommended fo determination of water in various drugs o Czechoslovak Pharmacopoeia containing v oils, since it avoids the errors caused in procedures by drying the material at 100° to The water in the sample is extracted into methanol by stirring for 5 min. in a closed v and is then titrated in the usual way.

J. Z

4561. **Study of the cation-exchange propert bentonite with application to the assay of pha ceuticals.** W. G. Gorman and E. P. Guth State Univ., Coll. of Pharm., Columbus). *J. Pharm. Ass., Sci. Ed.*, 1959, **48** (1), 21–26. acid-activated fraction of bentonite was exch saturated with gentian violet. This comple used for a study of exchange reactions with o cations. The amount of dye released fro complex, measured absorptiometrically, is a tion of the concn. of organic cations in soln could form the basis for their assay.

A. R. Rog

4562. **Toxicological investigations of micro q ities of alkaloids by ring paper chromatogr** Swarup Narain Tewari and Dharam Narain pathi (Clin. Lab., The Mall, Kanpur, I *Z. anal. Chem.*, 1959, **166** (5), 357–358 (in Eng —The separation of various alkaloids extract alkaline soln. from viscera, vomit, stomach w and urine is carried out by circular paper chro graphy. A drop of the alkaloid dissolved i acetic acid is placed at the centre of the filte (Whatman No. 1, 18 cm diam.) and the carrying the solvent (*n*-butanol - acetic acid - w (5:1:2) is applied below the centre of the paper. The whole system is kept in a chamber saturated with the solvent vapours a The paper is dried after 30 min. Quinine, c dine, cinchonidine and papaverine are detect fluorescence, while other alkaloids are detect the fluorescence-quenching technique of Ho

hnson (*Nature*, 1949, **163**, 216). The spots vealed by spraying with a modified Dragen- oln. [0·85 g of $Bi(NO_3)_3$, 10 ml of acetic acid ml of H_2O mixed with 8 g of KI in 20 ml]; for spraying, 10 ml of this soln. is diluted ml of acetic acid and 25 ml of H_2O. This t produces a variety of shades of orange and th various compounds. B. B. BAUMINGER

3. **Determination of the quinine content of um, totaquine, liquid extract of cinchona and na bark with the aid of ion-exchange resin.** amp and J. A. Fresen (Rijksuniv., Utrecht). *Weekbl.*, 1959, **94** (2), 33–39.—The total ids are isolated from the sample by chromato- y on an ion-exchange column (Dowex 50-X2) OH form) and converted into a neutral soln. sulphates. This soln. is added to a column ng anion exchanger (Dowex 1-X2) (chromate and left for 12 hr. All the alkaloids except e are then eluted with H_2O and the quinine vered by elution with either *N* aq. NH_3 or mic acid in 96% ethanol. The quinine can e determined bromimetrically.

G. BURGER

4. **Reactions for the detection of cytisine in ic studies.** I. A. Marenich. *Ukr. Khim.* 1958, **24** (3), 388–390; *Trudy Khar'kovsk. atsevt. Inst.*, 1957, (1), 119–122; *Ref. Zhur.*, , 1959, (1), Abstr. No. 1045.—The crystallo- reactions of cytisine (**I**) with the usual reagents are studied and the optical constants e resulting crystals are determined. **I** forms onal crystals with a soln. of $KBiI_4$, dendrites soln. of Br in NaBr soln., and clusters of needles 5% aq. soln. of H_2PtCl_6. The sensitivities of eactions are 0·3, 1 and 3 μg, respectively. ptical constants of the sublimed crystals of the , obtained as prisms by the sublimation of **I** der's method at 63° and 1 atm., or at 41° and , are determined. The sensitivity of the tion is 0·9 μg. It is found that **I** may be guished crystallo-optically from atropine, reacts analogously to **I** with a soln. of Br in soln.; certain constants of the compounds of ne with this reagent are studied. A method oposed for the detection of **I** in biological ial in forensic investigations.

C. D. KOPKIN

5. **The behaviour of some volatile *Nicotiana* ids on distillation and their quantitative nination.** C. Pyriki and R. Müller (Inst. f. xforsch., Dresden). *Pharm. Zentralh.*, 1959,), 7–19.—Distillation with MgO and steam- ation with NaOH may be used to separate quantities of nornicotine, but not of ana- e, from an excess of nicotine. To determine quantities of nornicotine, the method of ff and Markunas (*cf. Anal. Abstr.*, 1956, **3**, is recommended. The recovery of total ids by steam-distillation depends on the ne and alkalinity of the soln. distilled.

A. R. ROGERS

6. **Microchemical detection of anabasine and ifferentiation from nicotyrine and nicotine.** ndri (Ist. di Chim. Agrar. dell'Univ., Bologna). *chim. Acta*, 1959, (2), 221–229 (in German).— asine gives characteristic crystals with $HBiBr_4$, , $HAuBr_4$ and H_2PtBr_6; $CdBr_2$ is used as a nt for the crystallographic differentiation of ne and nicotyrine; and $Co(SCN)_2$ forms with ne characteristic pink (hydrated) and blue drous) crystals. B. B. BAUMINGER

4567. **Method for the determination of the flavonoids in tobacco.** M. K. Mikhailov (Hochsch. f. Nahrungs-u. Genussmittelind. Plovdiv, Bulgaria). *Compt. Rend. Acad. Bulg. Sci.*, 1958, **11** (6), 493–496 (in German).—The method is based on the measurement of the yellow colour given by the flavonoids with $AlCl_3$. The flavonoids are extracted into $CHCl_3$ and then into ethanol, and a portion is treated with $AlCl_3$ and K acetate. The colour is measured in a Pulfrich photometer with the S42 filter. Results are reproducible to within about 5%, and agreed to within $\simeq$20% with those obtained by another method. Good results were also obtained by aq. extraction of the tobacco.

G. P. COOK

4568. **Paper-chromatographic detection of theophylline and its water-soluble derivatives in pharmaceutical preparations.** H. R. Hegi (Kantonsapotheke, Zürich, Switzerland). *Pharm. Acta Helv.*, 1959, **34** (2), 105–109.—Descending chromatography on Whatman No. 1 paper with the upper layer of a mixture of *n*-butanol - 2 *N* aq. NH_3 (7:3) as mobile phase is used for the separation of 100 to 400-μg quantities of theophylline-7-acetic acid, theophylline, and the dihydroxypropyl, hydroxyethyl and hydroxypropyl derivatives of theophylline. The R_F values are 0·10, 0·30, 0·51, 0·65 and 0·71, respectively. Colour reactions with diazotised sulphanilic acid and diazotised 4-benzamido-2:5-diethoxyaniline (Fast blue salt BB) (C.I. Azoic Diazo Component 20) and the murexide test are used for detection. A. R. ROGERS

4569. **Determination of curcumin in drugs.** K. H. Müller and H. Honerlagen (Pharm.-Sci. Section II, A. Natterman and Co., Köln-Braunsfeld). *Arzneimittel-Forsch.*, 1959, **9** (2), 134.—The photometric method described is based on the formation of rubrocurcumin, a 1:1:1 complex of curcumin, boric acid and oxalic acid. *Procedure*—Heat 100 mg of finely powdered sample with 60 ml of acetic acid for 1 hr. at 90°, add 2 g of boric acid and 2 g of oxalic acid and heat for 10 min. Cool to room temp. and dilute to 100 ml with acetic acid. Dilute a 5-ml aliquot to 50 ml with acetic acid and read the extinction at 540 mμ. A. G. COOPER

4570. **Method for the assay of insulin by paper chromatography.** E. L. Fenton (Wellcome Res. Lab., Biol. Div., Langley Court, Beckenham, Kent, England). *Biochem. J.*, 1959, **71** (3), 507–513.—Insulin is separated from impurities and inactive material by ascending chromatography with the upper phase of *sec.*-butyl alcohol and 1% acetic acid (1:1, v/v). A soln. of bromocresol green buffered with Na acetate is used to locate the insulin zones. There is a linear relationship between the amount of insulin in the zone and the biological potency of the sample. By elution of the dye from the specific insulin spot and quant. colorimetric comparison with the similar zone from a standard of known potency, the biological potency of the sample can be determined. There is close agreement between the mouse-convulsion method and the chromatographic assay. The potency ratio of a single observation each on test and standard has a coeff. of variation of $\pm$4·3%. J. N. ASHLEY

4571. **Colorimetric estimation of penicillamine.** Prabhat R. Pal (Dept. of Biol. Chem., Univ. of Michigan, Ann Arbor, U.S.A.). *J. Biol. Chem.*, 1959, **234** (3), 618–619.—A photometric method is described which depends on measurement of the

blue colour formed when penicillamine is heated with $FeCl_3$ and KCN at 65° for 5 min. Measurement is made at 645 mμ and the amount of the amino acid is ascertained from a standard graph. The oxidised form of penicillamine does not react, cysteine gives only a very faint colour (5 to 7% of that given by penicillamine), and reduced glutathione does not give any colour under the specified conditions. Krebs - Ringer bicarbonate buffer and 0·25 *M* sucrose do not affect the colour formation. The method is applicable to the analysis of penicillamine in Ehrlich ascites tumour cells and other tissues if the extracts are deproteinised by heating at pH 5 to 6 and 100° for 10 min. J. N. Ashley

4572. **Photometric determination of ketonic substances in chloramphenicol.** A. Šlouf (State Inst. for Control of Drugs, Prague). *Československ. Farm.*, 1959, **8** (2), 77–79.—The method is based on the reduction by the ketonic group of a mixture of tungstophosphoric and molybdophosphoric acids (Folin - Ciocalteu reagent) to molybdenum blue. *Procedure*—To 0·1 g of the sample add ethanol (1 ml) and H_2O (1 ml) and dissolve by heating on a water bath. Add the reagent (1 ml), H_2O (10 ml) and Na_2CO_3 soln. (20%) (1·5 ml) and heat for 5 min. on a boiling-water bath. Cool, dilute to 25 ml, filter, and reject the first 5 ml of filtrate. Measure the extinction of the succeeding filtrate at 610 mμ and compare with a calibration curve; 50 μg of ketonic compound can be determined in 0·1 g of chloramphenicol. *Reagent*—Dissolve $Na_2WO_4.2H_2O$ (50 g) and $Na_2MoO_4.2H_2O$ (12·5 g) in H_2O (350 ml), add H_3PO_4 (85%) (25 ml) and HCl (36%) (50 ml) and heat under reflux for 10 hr. Add Li_2SO_4 (75 g), H_2O (25 ml) and bromine (3 drops). Remove the excess of bromine by boiling, and dilute to 500 ml. J. Zýka

4573. **Translocation of antibiotics in higher plants. III. Estimation of griseofulvin relatives in plant tissue.** S. H. Crowdy, A. P. Green, J. F. Grove, P. McCloskey and A. Morrison (I.C.I. Ltd., Akers Res. Lab., Welwyn, Herts., England). *Biochem. J.*, 1959, **72** (2), 230–241.—Methods similar to those used for the determination of griseofulvin (Crowdy *et al.*, *J. Exp. Bot.*, 1955, **6**, 371) are used for the determination of 11 compounds related to griseofulvin in bean tissue and in treating soln. They are determined spectrophotometrically after extraction from the tissues or soln. and purification by counter-current distribution and/or chromatography. J. N. Ashley

4574. **Polymorphism and changes of infra-red spectra of barbiturates during sample preparation.** B. Cleverley and P. P. Williams (Dominion Lab., D.S.I.R., Wellington, N.Z.). *Chem. & Ind.*, 1959, (2), 49–50.—Crystalline barbiturates which contain no hydroxyl groups show spectral changes whether prepared as mulls or as potassium bromide discs; X-ray studies have shown that these changes are due to the formation of another crystalline form. This polymorphic change is induced by grinding and pressing and the presence of KBr has no effect. K. A. Proctor

4575. **Note on the determination of dibasic amphetamine phosphate in the presence of methyl cellulose and barbiturate.** W. M. Studebaker and S. M. Wang (Southern Coll. of Pharm., Atlanta, Ga., U.S.A.). *Drug Standards*, 1959, **27** (2), 41.—Transfer the sample (containing 200 mg of dibasic amphetamine phosphate) to a Kjeldahl flask with H_2O (200 ml), add 50% NaOH soln. (5 ml) and anti-foam agent Velvacil 1000 (5 drops) and distil into 0·02 *N* H_2SO_4 (20 ml). Wash the condenser with 0·02 *N* H_2SO_4, dilute the combined distillate and washings to 200 ml and measure the extinction at 258 mμ. A. R. Roge

4576. **Determination of Largactil [chlorpromazine].** P. Spacu, E. Antonescu and C. Gheorghiu (Acad. Centr. Res., Bucharest). *Acad. R.P.R. Stud. Cercet. Chim.*, 1958, **6** (4), 573–584.—conditions for gravimetric macro- and micro-determinations based on the use of ammonium reineckate are given. The acetone-soluble violet compound formed by chlorpromazine with $K_3[Cr(SCN)_6]$ is used in a spectrophotometric method, the details of which are given. Details are also given for a method based on pptn. with **I** and potentiometric titration of the $[Cr(SCN)_6]^{3-}$ with $AgNO_3$ soln. H. Sh

4577. **Determination of pentazol [leptazol].** P. Spacu, I. Albescu and C. Gheorghiu (Acad. C Res., Bucharest). *Acad. R.P.R., Stud. Ce Chim.*, 1958, **6** (4), 565–572.—A new com $(C_6H_{10}N_4)H[Cr(NH_3)_2(SCN)_4]$ (**I**) from the int tion of leptazol with ammonium reineckate is basis of the three proposed methods. (*i*) *Spe photometric determination*—A soln. (≃10%) of sample is adjusted to pH 1·5 with conc. HCl. chilled soln. is treated with a soln. of ammon reineckate (2·5%), containing 10% of conc. until there is a slight excess. After a few min the ppt. of **I** is filtered off and the excess of reagent is removed by a mixture of benzene (10 ether (5 drops) and dioxan (2 drops). The pp dissolved in acetone and determined in a Pul spectrophotometer, with a green filter; the absorption is at 533 mμ. Amounts from 29 μ 2 mg were determined with errors within the li of the apparatus. (*ii*) *Volumetric determinati* This method is based on the formation $Ag[Cr(NH_3)_2(SCN)_4]$ (**II**) and titration of excess of Ag^+ with NH_4SCN. A soln. of sample (⩾10% of leptazol) is treated with HNO_3 to pH 1·5 to 1·6. Ammonium reinecka added in slight excess to the chilled soln. and a few minutes the ppt. is filtered off, the exce the reagent being removed with the solvent mix as before. The ppt. is dissolved in acetone treated with an excess of $AgNO_3$. The ppt is filtered off and an aliquot of the filtrate is tit with 0·1 *N* NH_4SCN, with $(NH_4)_2SO_4.Fe_2(SO_4$ indicator. (*iii*) *Potentiometric determination*— excess of Ag^+ is determined potentiometrical room temp., with a silver wire - normal cal electrode pair. H. Sh

4578. **Determination of chloroform in aqu pharmaceutical preparations.** L. Brealey, D Elvidge and K. A. Proctor (Standards Dept., Pure Drug Co. Ltd., Beeston, Nottingham, land). *Analyst*, 1959, **84**, 221–225.—A gas-chr tographic method is described. By means c injection pipette system based on that of Te and Harris (*Anal. Abstr.*, 1957, **4**, 2843), described in detail, the sample is injected by th stream into a cup containing glass wool to tra non-volatile components. The block suppo the cup and containing the gas inlet is he electrically and the column, with a the conductivity detector, is placed in an oil The stationary phase is a high-mol.-wt. ethylene glycol, *viz*, Carbowax 1500. Since

s are thick suspensions or syrups, an internal rd procedure is necessary and *n*-propanol is ecause its peak appears between those of and water. Calibration graphs are obtained asuring the ratio of the peak heights of and *n*-propanol, and standards containing ? and 0·4% of $CHCl_3$ and 1% of *n*-propanol er are used. Standards and samples are tographed under specified conditions with carrier gas. Quoted results indicate satis- accuracy. A. O. JONES

. **Colorimetric assay for camphor in camphor** M. I. Blake and L. V. Hopkins (N. Dakota Coll., Sch. of Pharm., Fargo, U.S.A.). *Drug rds*, 1959, **27** (2), 39–40.—Dissolve *p*-di- aminobenzaldehyde (0·125 g) in H_2SO_4 (65 d H_2O (35 ml). Add the reagent (7 ml) to nple (40 μl), shake, and measure the extinction mμ after 2 hr. The procedure is much more han the gravimetric assay of the U.S. National lary and is almost equally accurate and . A. R. ROGERS

. **Polarographic determination of cobalt, n B_{12} [riboflavine] and vitamin C [ascorbic acid] es in tablets and ampoules.** M. Sterescu, zan and M. Popa. *Rev. Chim. Bucharest*, **10** (2), 109–111.—Cobalt is determined by -reduction of the hexa-amminocobaltic ion xidation by sodium perborate in ammoniacal Under these conditions riboflavine and ic acid are destroyed, and the polarographic ination of Co proceeds normally. Ribo- is extracted from the sample with an aq. f Na salicylate (5%), and is then determined graphically. Ascorbic acid is determined dilution of the sample with acetic acid - buffer soln. (pH 4·6). It was established e other ingredients of the tablets or ampoules interfere, and errors are $\gtrless \pm 2\%$. H. SHER

. **Note on the assay of ethyl aminobenzoate caine] ointment N. F. X.** P. J. Levine and Allen (Sch. of Pharm., Univ. of Maryland, lore, U.S.A.). *Drug Standards*, 1959, **27** (2), issolve the sample in light petroleum and with several portions of aq. HCl. Cool the ned aq. phases in ice and titrate the benzo- with $NaNO_2$ soln. The recovery of benzocaine ntitative. A. R. ROGERS

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also Abstracts—**4446**, Determination of acetyl . **4460**, Refractive index of dextran. **4464**, on of sulphanilamide. **4479**, Reactions of yclic triterpenes. **4480**, Determination of ic acid deriv. **4523**, Catecholamines in **4524**, **4525**, Reaction of adrenaline with nediamine. **4542**, Determination of pure gens. **4589**, Analysis of malt extract. **4597**, nination of vitamin A in oily soln. **4601**, nination of "nerve gases" in air. **4614**, of chlortetracycline in feeds.

Food

oods and food additives, beverages, edible oils and fats, vitamins.

. **Chromatographic detection of hydrolysed syrup in adulterated natural honey.** M. chowicz and A. Andrełowicz (Dept. of Food eeding Hygiene, District San.-Epidemiolog. Stat., Bydgoszcz). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 259–264.—Descending chromatography is carried out on Whatman No. 1 paper with the solvent system *n*-propanol - ethyl acetate - water (6:1:3). The separated components are revealed by treating the chromatogram with an acetone soln. of $AgNO_3$, drying, dipping in an ethanolic soln. of NaOH, then in aq. NH_3, washing in H_2O, immersing for 10 min. in a 15% soln. of $Na_2S_2O_8$, washing again in water and drying in air. Although the use of the solvent system named does not enable glucose and fructose to be differentiated, a constituent characteristic of natural honey, and which does not occur in hydrolysed sugar syrup, is separated (R_G 0·47). The time of development is $\simeq$44 hr. the method is particularly useful for the serial examination of suspected samples. W. B. MIASKOWSKI

4583. **Determination of the apparent purity of beet sugar factory juices and syrups. II.** W. H. Parker (British Sugar Corp., Ltd., 134, Piccadilly, London). *Int. Sugar J.*, 1959, **61**, 9–13.—The conductance of an impure sugar soln. is practically constant at a density corresponding to $\simeq 28° \pm 2°$ Brix; soln. therefore need not be diluted to exactly 28° Brix for making conductivity measurements. An automatic diluter is described which adjusts the sugar soln. to 28° Brix and then discharges it into a cooling bath which adjusts the temp. to 20°. The soln. is then run into a copper bath, in which are suspended brass tubes containing standard sugar soln. for comparison. The electrode cell forms part of an unbalanced bridge, and conductivity measurements are taken by immersing the cell first in the standards and subsequently in the soln. under test. E. DUX

4584. **Determination of carbon dioxide in molasses.** F. Durdík. *Listy Cukr.*, 1959, **75** (1), 10–11.—The vol. of the foam formed on acidifying the molasses has been compared with the CO_2 measured manometrically in a Warburg apparatus. It is stated that, in some varieties of molasses, part of the CO_2 is already liberated by the dilution with H_2O and a correction factor must therefore be used in the calculation of the manometric results. J. ZÝKA

4585. **Amperometric determination of mercapto groups in grain proteolysis.** G. I. Kotlyar (Lvov Trade-Econ. Inst., USSR). *Biokhimiya*, 1959, **24** (1), 15–18.—The determination was carried out with 0·001 *N* $AgNO_3$ (1 ml) in an ammoniacal medium (50 ml) containing 0·2 *N* aq. NH_3 and 0·2 *N* NH_4NO_3 (pH 9·37). The soln. was kept free from O by saturating it with N both before and during the titration. A rotating platinum electrode was used. The decrease in diffusion current after the addition of the aq. extract (1 ml) of the powdered grain was directly proportional to the decrease in concn. of Ag^+ as a consequence of the pptn. of the mercapto groups. The mercapto groups were assayed in aq. extracts of powdered grain without infusion and after a 2- and 4-hr. autolysis. Back-titration was employed to prevent both oxidation of the mercapto groups and their partial combination with NH_4^+ owing to the high pH of the ammonia buffer. Aq. extracts and protein-free filtrates of wheat, rye and barley both before and after a 96-hr. germination were investigated. The mercapto-group content increased in all cases after a 4-hr. autolysis; this was particularly marked in germinated grains. K. R. C.

4586. **Spectrophotometric determination of cholesterol in "pasta all'uovo."** F. De Francesco and A. Franciosi (Lab. Chim. Provinc., Trento, Italy). *Boll. Lab. Chim. Provinciali, Bologna*, 1959, **10** (1), 57–63.—*Procedure*—Milled "pasta" (10 g) is extracted with diethyl ether for 4 hr. in a Soxhlet apparatus. After evaporation of solvent, 10 ml of a 20% KOH soln. in 70% ethanol is added, and saponification carried out. The hydrolysed soln. is transferred to a separating-funnel with H_2O (3 × 50 ml) and extracted with diethyl ether (3 × 10 ml). The ether extracts are combined and washed with 15 ml of 1% KOH soln. and then with water (3 × 5 ml). The ether layer is dried with anhyd. Na_2SO_4, then filtered and evaporated to dryness. The residue is made up to 100 ml with the reagent (0·05% $FeCl_3$ in glacial acetic acid). An aliquot of this soln. (0·5 ml) is made up to 5 ml with reagent, 3 ml of conc. H_2SO_4 is added and the extinction read after 20 min. The resulting violet colour is very stable and shows an absorption max. at 560 mμ. The results are almost free from errors due to other sterols. L. Zanoni

4587. **Experiments on the detection of preservatives using the agar diffusion test.** F. Baum and H. Lamm (Inst. Ernährung, Potsdam-Rehbrücke, Berlin, Germany). *Ernährungsforschung*, 1959, **4** (1), 84–89.—The actions of benzoic, dehydroacetic and sorbic acids against the test organisms (*Rhodotorula* spp., *Penicillium* spp., *Aspergillus niger*) and baker's yeast were followed by growth on agar plates. The width of the inhibition zones for a given quantity of preservative is specific for each preservative. J. V. Russo

4588. **Chromatographic studies of the artificial colourings of foodstuffs. I. Column chromatography.** V. Sadini (Ist. di Merceolog., Univ. di Trieste, Italy). *Chim., Milan*, 1959, **35** (1), 15–26.—A review is given of methods for determining dyes in foodstuffs, with 115 references.

II. Paper chromatography. V. Sadini. *Ibid.*, 1959, **35** (2), 81–98.—A review is given of methods for determining dyes by paper chromatography, with 158 references. L. Zanoni

4589. **Method of assessment of the genuineness of malt extract.** F. Muntoni and A. Cesari (Ist. Sup. Sanità, Roma, Italy). *Boll. Lab. Chim. Provinciali, Bologna*, 1959, **10** (1), 3–11.—*Procedure*—Malt extract (10 g) is dissolved in 5 ml of boiling water. After cooling, 130 ml of 95% ethanol is added to precipitate the dextrins and the mixture is set aside for 24 hr. The ethanolic soln. is decanted, the ppt. is dissolved in 5 ml of boiling H_2O, and the dextrins are re-pptd. and the mixture is set aside for a further 24 hr. The combined ethanolic extracts are distilled at 200 mm of Hg at 65° to 70° to remove the ethanol. The residue is transferred to a 100-ml flask and 5 ml of 10% (w/v) basic Pb acetate and, after 10 min., 10 ml of satd. Na_2SO_4 soln. are added. The soln. is made up to volume, then filtered, and the polarimetric reading is made in a 2-dm tube after 1 hr. An aliquot (20 ml) of the filtered soln. is diluted to 100 ml and the reducing power with Fehling's soln. (diluted 1:4) is determined. Then maltose (%) = $[202{\cdot}2\ P - (5280/R)]/41{\cdot}634$, and glucose (%) = $[(13820/R) - P]/41{\cdot}634$, where R is the vol. (ml) of sugar soln. required to reduce 10 ml of Fehling's soln. and P is the polarisation in a 2-dm tube. The maltose to glucose ratio is never less than 3·20 for genuine malt extracts. L. Zanoni

4590. **Use of the anthrone procedure i carbohydrate analysis of beer.** G. J. Haas and Fleischman. *Wallerstein Labs Commun.*, 195 139–151.—Hexose polysaccharides are hydrolysed by strong H_2SO_4 in the presen anthrone to give a green colour. The proc based on this reaction is not affected by s factors that are troublesome in other me *Procedure*—De-gassed beer is diluted with (1:250). The diluted beer (1 ml) and water are placed in a test-tube and 10 ml of 0·1% ant in H_2SO_4 (76% by vol.) is added. The mixt shaken, heated in a boiling-water bath for 12 then cooled to room temp. The green developed is read in a photo-electric colori with a suitable filter, correction being made f reading obtained on a water - reagent blank. standardisation, determinations are run in the way on a 1% aq. glucose soln. There is a relationship between glucose concn. and intensity. Tannic acid and various amino have no interfering action, and the presen pentose causes only a negligible error provide its concn. is <10% that of the hexoses. values obtained by this procedure for total hydrates expressed as glucose are slightly than by other methods based on acid hydr owing to the loss of reducing groups in the cases. J. Inst. Brew. Abs

4591. **Differential thermal analysis of fat Principle, apparatus and procedure.** A. J. Hai and J. Hannewijk (Unilever Res. Lab., Vlaard Netherlands). *J. Amer. Oil Chem. Soc.*, 19 (7), 344–347.—The m.p. and polymorphic prop of various fats and oils were studied by differ thermal analysis; the curves obtained were characteristic for each substance. The app is fully described.

II. Melting behaviour of some pure glyc H. Lavery (Unilever Res. Dept., Port Sun England). *Ibid.*, 1958, **35** (8), 418–422. melting characteristics and polymorphic trans of 7 mono-oleyl disaturated glycerides wer amined by X-ray and differential thermal an results found by other workers were largely firmed. The unstable α-forms of the glyc were obtained by shock chilling, and other by heating or recrystallisation from a so most of these were confirmed by X-ray an Each of the 7 melting curves was distinctive. influence of pre-melting on the apparent lower the initial m.p. is discussed. P. D. Parr-Rich

4592. **The study of animal fats in vegetabl** G. Bigoni (Soc. Gaslini, Genova, Italy). *Olii* 1959, **36** (1), 1–4.—The method is based o separation of the solid fatty acids as Pb and distillation of part (≃ 15%) of the r esters prepared from the liquid fraction specified pressure and temp. The mean sap. of the distillate is determined. The esters olive oil have a mean sap. value of < 1 animal fats are present (> 10%) this value from 194 to 212. A new value is proposed "lower liquid acid value" (LLAV) = (SN − 100, where S is the sap. value and P the perc of distilled ester. This value varies from − + 0·15, and increases when animal fats are p Coconut oil increases this value, but this detected by determination of the Wollny Polenske numbers. L. Zan

Applications of spectrophotometry in the of olive oil. I. Use of ultra-violet spectro- etry for the characterisation of virgin olive . Montefredine and L. Laporta (Lab. Chim. i Pescara, Italy). *Olii Min.*, 1959, **36** (2), –The ultra-violet spectra of various types of l, and the effects of oxidation, heat and treatments on the absorption in the 270-mμ are discussed. L. A. O'NEILL

Determination of tocopherol in oxidised nterference from heat-formed reducing sub- in highly oxidised fats. E. N. Frankel, P. M. , C. D. Evans and J. C. Cowan (U.S. Dept. Peoria, Ill.). *J. Amer. Oil Chem. Soc.*, **5** (11), 600–602.—In the determination of erols in oxidised fats, the peroxides can be iently removed by heating the sample at *vacuo* for 15 min. if the peroxide levels hin the range 0 to 100 (by the ferric thio- e method). By a chromatographic procedure h 1 g of oil in 10 ml of benzene is passed a column (20 mm diam.) containing 1 g ic acid, and eluted with 50 ml of benzene removes the peroxides), it is shown that the oduced reducing substances have little n the tocopherol determination within the ed peroxide range. The presence of such ces causes an increase in darkening, vis- acidity and carbonyl values, with a decrease e value. G. R. WHALLEY

Detection and measurement of *cis*-unsatura- fatty acids. R. T. Holman, S. Euer and P. R. dson (Minnesota Univ., Austin, U.S.A.). *Biochem. Biophys.*, 1959, **80** (1), 72–79.—The is based on measurement of the extinction 0% soln. of the fatty acids isolated from oils in CCl_4 at 2·15 μ by a base-line tech- At this wavelength, *cis*-unsaturation only rmined. Fatty acids with other types of ration do not interfere, and calibration is with linoleic - palmitic and with oleic - acid mixtures. Results are calculated from lowing expression—moles of *cis* per kg = $\times 10^4) - 0{\cdot}96$, where k is observed extinc- r g of sample per litre. Good agreement is d with results for *cis*-unsaturation obtained he iodine value corrected for *trans*-unsatura- easured at 10·36 μ. W. H. C. SHAW

. Analytical application of iron(III) hydrox- . II. The determination of the esters of ed, unsaturated, straight-chain and branched cids from C_2 to C_5. W. Pilz (Organ.-anal. . Farbenfabrik. Bayer A.-G., Leverkusen). *. Chem.*, 1959, **166** (3), 189–193.—Previous n the determination of esters as ferric hydrox- (*cf. Anal. Abstr.*, 1959, **6**, 820) has been ed to a wide range of esters. It is shown e extinctions of the ferric hydroxamate soln. mμ are approximately the same for equal of ester in milli-equiv. per ml. T. R. ANDREW

. Determination of vitamin A in oily solution. inger (Lab. Physiol., Acad. Sci., Ivanka Pri , Czechoslovakia). *Farmácia, Bratislava*, **8** (2), 44–50.—The losses ($\simeq 7\%$) of vitamin ing its quant. determination, due to the ce of light and air, have been studied. Most oss occurred during the extraction. The use glass and an inert atmosphere during treat- vith KOH prevents the errors. *Procedure*— of the oil add ethanolic KOH soln. to a concn. of 0·5 *N*. Boil for 15 min. under reflux in an atmosphere of N, then extract four times with peroxide-free diethyl ether; dry the extract with anhyd. Na_2SO_4, evaporate an aliquot in a stream of CO_2, dissolve the residue in spectrally pure ethanol and determine the extinction at 311, 326·5 and 334 mμ. J. ZÝKA

4598. **Qualitative and quantitative colour reaction for pyridoxal with sulphuric acid.** V. E. Levine and R. N. Sass (Creighton Univ. Sch. of Med., Omaha, Nebr., U.S.A.). *Anal. Chim. Acta*, 1959, **20** (2), 137–146.—Phenols react with aldehydes and ketones, under the influence of a condensing agent such as HCl or H_2SO_4, to give a coloured product. The analytical applications of this type of reaction are discussed and its sensitivity for various phenolic aldehydes and phenolic ketones is determined. Of the four phenolic compounds related to vitamin B_6, pyridoxol, pyridoxamine, pyridoxal (**I**) and pyridoxic acid, **I** is the only one that is also an aldehyde. Consequently the intense yellow colour that **I** yields with conc. H_2SO_4 provides a selective method for its detection in the presence of the other three compounds. The reaction is sensitive to 10 μg when **I** is heated with H_2SO_4 on a water bath for 10 to 15 min. The addition of a trace of $CuSO_4$ increases the sensitivity to 1 μg. When 10 to 100 μg of **I** in 1 ml of water is mixed with 4 ml of conc. H_2SO_4 at room temp., the resulting colour, measured at 390 mμ, follows the Beer - Lambert law. A second absorption peak occurs at 335 mμ. A short review is given of analytical methods for the determination of the B_6 group and pyridoxic acid. H. N. S.

4599. **Assay and distribution of bound forms of pantothenic acid.** G. M. Brown (Div. of Biochem., Dept. of Biol., M.I.T., Cambridge, U.S.A.). *J. Biol. Chem.*, 1959, **234** (2), 379–382.—A microbiological method is described for the determination of pantothenic acid, phosphopantothenic acid, pantetheine, phosphopantetheine, and coenzyme A in any one material. It depends on the differential enzymic degradation of coenzyme A and phosphopantetheine to give pantetheine, and the degradation of phosphopantothenic acid to pantothenic acid. Pantetheine is determined first on the sample by use of *Lactobacillus helveticus* and then, after the action of prostatic phosphatase, *Lb. helveticus* determines pantetheine plus phosphopantetheine. The same organism, after the action of intestinal phosphatase, determines the sum of pantetheine, phosphopantetheine, coenzyme A and dephospho-coenzyme A. Pantothenic acid is determined in the sample by means of *Saccharomyces carlsbergensis*, and this organism, after the action of prostatic or intestinal phosphatase, is used to determine pantothenic acid plus phosphopantothenic acid. Coenzyme A and dephospho-coenzyme A cannot be separated by this method. J. N. ASHLEY

4600. **Recent methods of vitamin C [ascorbic acid] determination.** L. Káplár (Gen. Chem. Inst., Polytech. Univ., Budapest). *Magyar Kém. Lapja*, 1959, **14** (2–3), 122–126.—Methods, in particular those published since 1945, are reviewed. (74 references.) G. SZABO

See also Abstracts—**4456**, Hydroperoxides in oils. **4458**, Effect of sodium borate on optical rotation of sugars. **4484**, Determination of starch. **4510**, Determination of sugars in fruits. **4580**, Determination of riboflavine and ascorbic acid in mixtures.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

4601. **Sampling and analysis of toxic vapours in the field.** E. Neale and B. J. Perry (Chem. Defence Expt. Estab., Porton Down, Salisbury, Wilts., England). *Analyst*, 1959, **84**, 226–232.—The colorimetric determination of the "nerve gases," *e.g.*, Tabun (ethyl dimethylphosphoramidocyanidate) and Sarin (*iso*propyl methylphosphonofluoridate), depends on their reaction with H_2O_2 and an aromatic base of the benzidine type in alkaline soln. In the original field method, a measured amount of air was drawn through *cyclo*hexanol in a spined bubbler. The sample was then adjusted to 40 ml with a phosphate buffer (pH 5·5) and a suitable aliquot was treated with H_2O_2 and *o*-dianisidine soln. The pH was then raised to 11·0 by an addition of another buffer soln., the vol. was adjusted to 25 ml with an acetone - water mixture and the colour was measured in an absorptiometer. In the apparatus now described, all these operations can be effected without removal of the sample from the specially designed bubbler, the normal range being covered by the use of 0·25 to 3-cm cells in the absorptiometer. The procedure is applicable also to field measurements of Cl, $COCl_2$, CNCl and mustard gas, and probably for many other volatile toxic substances. A. O. Jones

4602. **Determination of total gaseous pollutants in atmosphere.** P. W. West, Buddhadev Sen and Bharat R. Sant (Louisiana State Univ., Baton Rouge, U.S.A.). *Anal. Chem.*, 1959, **31** (3), 399–401.—The method is based on the measurement of the thermal conductivity when small quantities of organic solvents or inorganic gases are introduced into a stream of He flowing through an empty column; a symmetrical distribution of the concn. of the solutes occurs by diffusion. The times of emergence and the areas under the curves are reasonably the same for the same quantity of a single solute or for mixtures, despite the possible differences in physical properties. Ten compounds were tested and the total error rarely exceeded 20%. G. P. Cook

4603. **Measurement of dissolved oxygen in swamp waters. Further modification of the Winkler method.** L. C. Beadle (Makerere Univ. Coll., Kampala, Uganda). *J. Exp. Biol.*, 1958, **35** (3), 556–566.—Errors in the Winkler iodine-difference method, which are especially large with highly polluted waters, are overcome by making a preliminary clarification of a single sample from which two smaller samples are siphoned, and by the addition of iodine with the clarifying reagents to the original sample. An improved iodine titration end-point is described involving the use of a magnetic stirrer. Direct spectrophotometric determination of iodine is shown to be as accurate as, and very much more rapid than, titration, and particularly suited to the iodine-difference method. The modified method is accurate to within 0·2 mg of O per litre. With polluted water, having an oxidation - reduction potential of $< +100$ mV, 'negative' figures for the oxygen content are obtained. The cause of this is unknown but, since this potential indicates the absence of free O, the method is not thereby affected. K. R. C.

4604. **Conductimetric analysis of natural II. Determination of magnesium and anions.** Pasovskaya (Turkmen Medicinal Inst., Ashkl *Zhur. Anal. Khim.*, 1959, **14** (1), 108- *Determination of Mg*—Neutralise 5 to 25 ml water to methyl red with 0·1 *N* HCl, evap necessary to 5 or 10 ml, adding more acid if sary but avoiding excess, cool to 80°, add 1 satd. ammonium oxalate soln. and 0·5 ml of soln. of pH 10 (0·1 *N* aq. NH_3 - 0·1 *N* NH_4Cl dropwise with stirring to precipitate Fe(OH $Al(OH)_3$ (which do not interfere), cool to temp., dilute to 50 ml and titrate conductime with EDTA (disodium salt). The results ob agree with those of the gravimetric pyropho method. *Determination of sulphates*—Neu the water (2 to 10 mg of SO_4^{2-}) to methyl re 0·1 *N* HCl, add dry ground $BaSO_4$ ($\simeq$100 m 0·5 ml of satd. methyl violet soln., mix, add of standard 0·1 *N* $BaCl_2$, dilute to 100 ml and the excess of $BaCl_2$ conductimetrically w Na_2SO_4. *Determination of chlorides*—Neutral water (2 to 20 mg of Cl^-) to methyl red v HNO_3 and add 2 or 3 drops in excess, dilut ml and titrate conductimetrically with $Hg(NO_3)_2$. If a yellow cloudiness forms, p a fresh sample but add 6 or 7 drops of HI excess after the neutralisation. *Determina carbonate hardness*—In the absence of phos add to the water (0·02 to 0·2 milli-equiv. of dry $BaCl_2$ (100 mg), 0·01 *N* HCl (5 ml) and acetic acid (5 ml); if Fe is present add NaF soln. is colourless or replace the acetic aci 50 mg of citric acid; dilute to 50 ml and conductimetrically with 0·1 *N* $Ba(OH)_2$. If phates are present they are first removed by 5 ml of 1% $BaCl_2$ soln. and filtering after min. *Determination of total hardness*—Neu the water (0·03 to 0·06 milli-equiv. of Ca ar to methyl red with HCl, boil for several m adding more acid if necessary, cool to room dilute to 50 ml, add 0·5 ml of buffer soln. (p and titrate conductimetrically with 0·1 *N* (disodium salt). C. D. Ko

4605. **Field method for the determinati uranium in natural waters.** G. H. Smith and T Chandler (Chem. Res. Lab., Teddington, M England). Second U.N. International Conf on the Peaceful Uses of Atomic Energy, G 1958, Rep. A/CONF.15/P/298, 11 pp.—A method was developed for field use in geoch prospecting. It is based on the formati pH 6, of the yellow uranium - dibenzoylm complex which is extracted from the sampl CCl_4. The organic phase is separated fro water and its colour intensity compared wit of a set of standards. In this way it is p to detect amounts of U equiv. to 1 μg of U_3O_8 dibenzoylmethane is added to the water samp 0·5% (w/v) soln. in a 1:1 (v/v) pyridine - mixture containing 0·6% (w/v) of EDTA (dis salt) to complex interfering metal ions. As acid is also added to eliminate the interf otherwise caused by Fe^{3+}. It was found that waters contain organic matter that caused e fication and orange - brown colours in the layer. To remove this interference, several of adsorbent material were examined, inc silica gel, activated alumina, kaolin, pptd. and several grades of activated charcoal. Of certain grades of activated charcoal proved the most satisfactory. The adsorbent is ad the acidified water sample, which is then filter

ised with alkali before proceeding with the l. It was found that the addition of 2% by silicone fluid to the CCl_4 helped to prevent ication, as does the addition of NaCl to the ase. NUCL. SCI. ABSTR.

. **Determination of the phenol index of waters.** laminck (Centre Belge d'Ét. et de Document. ux, 2 rue Arm. Stevart, Liége). *Bull. Cent. Étud. et Docum. Eaux*, 1959, (43), 40–48.—ls for determining phenols in water and con- ing phenols present in water in low concn. cussed critically, and it is concluded that the uitable are extraction with diethyl ether from O_3 soln. and colorimetric determination by ng with diazotised *p*-nitroaniline. *Procedure* n non-phenolic extractable impurities are t, the water (200 ml) is made alkaline with of 4% NaOH soln. and the impurities are ed with ether, before proceeding with the d described. The sample of effluent water l) is acidified with H_2SO_4, and $NaHCO_3$ is until some remains undissolved. The phenols en extracted with ether and extracted back 0 ml of 4% NaOH soln. The ether is washed 0 ml of water, and the washings are added to aOH soln., which is then neutralised with and made up to 100 ml. The soln. (or the l water if no preliminary concentration is l out) is treated with 2 ml of diazotised *p*- niline (10 ml of 0·75% $NaNO_2$ soln. added to of a soln. containing 0·69 g per litre of *p*- niline in dil. H_2SO_4 immediately before use). xtinction is measured at 475 mμ within 1 hr. waters are similarly treated with the addition aric acid soln. to the sample. The prepara- nd standardisation with bromide - bromate of andard phenol soln. are described, and it is mended that they should be used within 2 hr. paration. It is recommended that samples of should be made alkaline if they are to be kept re than a few hours. E. J. H. BIRCH

7. **Radiochemical determination of radium-C horium-C in radioactive springs.** I. Hataye oka Univ., Japan). *Mem. Fac. Sci., Kyushu Ser. C*, 1958, **3** (2), 63–70.—The method was ed for the determinations of radium-C (^{214}Bi) d thorium-C (^{212}Bi) (**II**) in radon-containing ated mineral springs; **I** and **II** were extracted amyl alcohol as BiI_4^-, on the addition of KI l soln. The respective activities of extracted **II** were measured by a Lauritsen-type oscope; **II** was used as a tracer in the deter- ion of **I**. K. R. C.

8. **Radiochemical determination of radium-B horium-B in radioactive springs.** I. Hataye oka Univ., Japan). *Mem. Fac. Sci., Kyushu Ser. C*, 1958, **3** (2), 71–81.—The method was ed for determinations of radium-B (^{214}Pb) d thorium-B (^{212}Pb) (**II**) in radon-containing ated mineral springs; **I** and **II** were extracted soln. of dithizone in CCl_4 (50 mg per litre) an ammoniacal 5% KCN soln. at pH 8 to 9. event interference by Po and Bi, two elimina- nethods were proposed. It was found that extracted preferentially from a soln. containing es of Pb and Po when microgram quantities ctive Pb are added to the soln. Bismuth emoved by washing the extracts with 0·5% soln. after Pb and Bi had been extracted ter. The respective activities of extracted **I** and **II** were measured by a Lauritsen-type electroscope; **II** was used as a tracer in the determination of **I**. K. R. C.

4609. **Determination of anionic detergents in river water and sewage effluents.** J. G. Slack (Southend Waterworks Co., Treatment Plant and Lab., Ulting, Maldon, Essex, England). *Analyst*, 1959, **84**, 193.—A simplification of the method of Longwell and Maniece (*Analyst*, 1955, **80**, 167) is described, the time required for a determination being considerably reduced by extraction with only one 50-ml portion of $CHCl_3$. The reagents and apparatus are as previously described (*loc. cit.*), the $CHCl_3$ layer is run into the second separating-funnel, shaken, and filtered through cotton wool. After rejection of the first 5 ml the extinction of the filtrate is measured. Results agree satisfactorily with those obtained by the longer method.
A. O. JONES

4610. **Ultra-violet determination of the insect repellent diethyltoluamide.** C. H. Schmidt, M. C. Bowman and F. Acree, jun. (Entomology Res. Div., Agr. Res. Serv., U.S.D.A.). *J. Econ. Ent.*, 1958, **51** (5), 694–697.—The u.v. absorption spectra of *o*-, *m*- and *p*-diethyltoluamides in 95% ethanol are plotted between 215 and 285 mμ. There are no maxima in this region and the absorptions of the three isomers are different. Calibration curves (rectilinear) are prepared for the individual isomers at 230 mμ, at concn. of from 1 to 15 μg per ml. When studying commercial samples, which vary in the proportions of the isomers, a calibration curve must be prepared from the particular lot under investigation. The amount of diethyltoluamide deposited on glass or cloth is determined by washing with ethanol and measuring the absorption of the resulting soln. at 230 mμ. Extraction of cloth with ethanol yields a small amount of other material absorbing in the same region, and high recoveries are obtained unless the material has been deposited on ethanol-washed cloth. As there is no absorption max., a constant slit width (0·66 mm) is used. There is insufficient difference in the spectra of the three isomers to calculate the relative quantities by the absorption of the mixture at three wavelengths.
E. J. H. BIRCH

See also Abstracts—**4393**, Determination of Pu in water. **4399**, Determination of F_2O in air. **4506**, Determination of ^{89}Sr and ^{90}Sr in sea water. **4616**, Water sampler.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

4611. **Determination of water-soluble boron compounds in soil.** A. D. Gol'tman and V. G. Gurevich. *Trudy Khar'kovsk. Farmatsevt. Inst.*, 1957, (1), 110–114; *Ref. Zhur., Khim.*, 1959, (1), Abstr. No. 957.—The curcumin method is used. To 5 g of soil in a 25-ml flask add 10 ml of water, heat on a water bath at 50° for 30 min., shaking for 1 min. every 5 min., set aside for 20 min. at room temp. and filter. Determine B in an aliquot of the filtrate photometrically with curcumin. The presence of other substances in the aq. extract somewhat lowers the sensitivity of the determination of B; in the analysis of garden soil this error is 31%, but it is practically constant for various amounts of B

(0·2 to 1 μg) and may be allowed for. The reproducibility of the results is satisfactory; the deviation from the mean is ⩾1·5%. C. D. KOPKIN

4612. **Comparison of polarographic and colorimetric methods for the determination of nitrates in soil.** A. M. Artyushin and P. G. Kudelya. *Udobrenie i Urozhai*, 1958, (7), 20–24; *Ref. Zhur., Khim.*, 1959, (3), Abstr. No. 8000.—It is established that the polarographic method may be used for the determination of NO_3^- in a soil soln. or extract. Polarograph the soln. in a basal soln. of 0·1 *N* HCl and 0·00066 *N* uranyl acetate. Construct calibration curves from standard soln. The uranyl acetate gives two waves, the first at −0·181 V and the second at −0·909 V; NO_3^- form a wave on the second uranyl acetate wave; with increasing concn. of NO_3^- the reduction potential moves in the negative direction (from − 0·9 to − 1·1 V). Satisfactory results are obtained with a content of NO_3^- down to 350 mg per litre. The presence of organic substances in the soil does not influence the determination of NO_3^-. The polarographic results agree well with the colorimetric data, but the polarographic method is considerably more rapid. C. D. KOPKIN

4613. **Notes on the determination of potassium in fertilisers.** J. M. Rafols Rovira (Jefe Servicios Quím. de Aprovechamientos Salineros, S.A. Barcelona). *Inf. Quím. Anal.*, 1959, **13** (1), 14–17.—The cobaltinitrite method for the determination of K_2O in fertilisers is unsatisfactory for several reasons, but the perchloric acid method is reliable and gives reproducible results. G. H. FOXLEY

4614. **Problems in the assay of chlortetracycline in feeds.** A. E. Tanguay, A. B. Bogert and M. H. Blanchard (American Cyanamid Co., Pearl River, N.Y.). *Antibiot. & Chemother.*, 1959, **9** (3), 167–175.—Improved sensitivity and decreased interference from other constituents in commercial animal feeds are obtained in a three-level cylinder-plate assay for chlortetracycline (**I**) in animal feeds with *Bacillus cereus* var. *mycoides* ATCC 9634 as test organism. Satisfactory recovery and reproducibility were attained with ⩽20 g of sample extracted with acidified acetone. It is concluded that, for feeds containing >50 g of **I** per ton (U.S.), the use of a blank feed is unnecessary. At 20 to 50 g per ton, blanks are generally unnecessary, but may be prepared if required by autoclaving to destroy **I**. The effect of minerals in high-mineral feeds is considered, and methods for obtaining satisfactory recovery of **I** in such samples are discussed. W. H. C. SHAW

4615. **Modifications to a method for the determination of chlorinated hydrocarbon pesticide residues in plant material.** G. A. Sergeant and P. B. Thompson (Dept. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1959, **84**, 251–253.—The modified Stepanow procedure as previously described (*Analyst*, 1959, **83**, 335) is followed, but with ≃0·1 g of Na and 0·25 g of naphthalene, and 1·5 ml of *n*-butylamine in place of the tetrahydrofuran. After reaction, the excess of Na is removed by addition of 2 ml of aq. *iso*propyl alcohol (1:1) and the *n*-butylamine is removed by heating on the water bath; a few ml of water are added and then *p*-nitrophenol as indicator and enough dil. HNO_3 to render the liquid acid. The liquid is then extracted with ether, the aq. layer is separated and its vol. is reduced to ≃25 ml by heating. An aliquot (⩾20 ml), accurately measured, is treated with 2 ml of an $(NH_4)_2SO_4.Fe_2(SO_4)_3$ soln. (12 g and 40 ml of HNO_3 per litre) and then with 3 ml of eth $Hg(SCN)_2$ soln. (0·4%). After adjustment vol. to 25 ml the extinction is measured at 4 and referred to a calibration graph prepared KCl soln. treated with the colorimetric rea Quoted results with *pp'*-DDT, γ-hexachlor hexane, aldrin and dieldrin indicate satisf precision. A. O. Jo

See also Abstracts—**4353**, Determination of in soil extracts. **4515**, Assay of gibberellic ac

5.—GENERAL TECHNIQUE AN APPARATUS

General

4616. **Battery-operated water sampler.** Eden and K. V. Melbourne (Water Pollution Lab., Stevenage, England). *Chem. & Ind.*, (7), 220–222.—A portable unit for collecting sa of water from open channels at intervals of 3 to 1 hr. consists of a number of 12-oz bottles, of which is evacuated and connected by a r tube to the source from which liquid is to be d When a clamp on the tube is released electr netically, water is forced by air pressure int bottle. The construction and operation o automatic sampler, which contains the dry bat and timing unit, are described and illustrated. apparatus can be used only when the consti of interest in the sample is unaffected by stor the bottles. W. J. BA

4617. **Reduction of micro and semi-micro w ings to normal temperature and normal pre** C. J. van Nieuwenburg (Tech. Univ., Delft, N lands). *Anal. Chim. Acta*, 1959, **20** (2), 127–1 A simple apparatus (illustrated) yields a reading of the correction required to reduce and semi-micro weighings to an N.T.P. basis, compensating for buoyancy variations. H. N

4618. **A simple ultramicro-burette.** V. H mann (Inst. Med. Chem., Charles' Univ., P Czechoslovakia). *Chem. Listy*, 1959, **53** (1), 3 —A simple ultramicro-burette (illustrated) structed on the principle of the micrometer enables volumes to be delivered with an acc of 0·00024 ml. J. Z

4619. **Aspects of organic analysis. VII. free evaporation of solutions for crystallisation small scale.** G. W. Perold (Sect. for Coal an Chem., Sci. and Res. Dept., Iron and Steel Corp. Ltd., Pretoria, S. Africa). *Mikrochim.* 1959, (2), 251–253 (in English).—With the appa described, soln. (1 to 4 ml) such as a fraction f chromatographic separation or a soln. othe obtained can be evaporated or concentrated wi creeping. B. B. BAUMIN

Chromatography, ion exchange, electrophoresis

4620. **Detector for liquid - solid chromatogr** G. Claxton (Benzole Producers Ltd., Wa England). *J. Chromatography*, 1959, **2** (2), 139.—The detector consists of a section of jacketed capillary tubing packed with adsorb

a thermocouple is embedded. When a of composition occurs in the liquid leaving mn, a temperature change occurs in the owing to the change in the heat of adsorp- f the output from the thermocouple is , a peak is obtained when a component he column. W. T. CARTER

Improved scanner for radioactive paper F. Eisenberg, jun., and I. G. Leder (U.S. Health, Education and Welfare, Bethesda, S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), .—A simple, easily constructed automatic rip scanner is described, which gives a cible and strictly linear relationship between onding points on the chromatogram and K. A. PROCTOR

Calculations for a continuous gas-chromato- (hypersorption) column. L. Szepesy and dek. *Magyar Kém. Lapja*, 1958, **13** (10–12), .—The principles and formulae for calcula- e given for continuous charcoal columns for separation of gas mixtures into two or actions. Isothermal conditions are assumed number of theoretical plates, the places of nd reflux and the minimal velocity of char- e determined by the exchange equations uilibrium and adsorption constants. The ion of an H - CO_2 - acetylene mixture is ed. G. SZABO

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lso Abstract—**4433**, Catalytic hydrogenation er in paper chromatography.

Optical

High-temperature X-ray diffractometer. J. borough and J. W. Christian (Dept. of rgy, Oxford). *J. Sci. Instrum.*, 1959, **36** –118.—Constructional details are given of a ometer vacuum-furnace using a tantalum gh-current heater and capable of operating . up to 1100°. The temperature is measured platinum/platinum - rhodium thermocouple n be controlled to $\pm 2°$. The instrument the full Bragg spectrum to be recorded while ing the Bragg - Brentano focusing condition. G. SKIRROW

. Generally applicable semi-quantitative chemical techniques. H. Svejda (Staatl. -tech. Versuchsanst., Vienna). *Öst. Chem-*959, **60** (2), 37–44.—A review, with 25 refer- E. G. CUMMINS

. Illumination in spectrometers. M. Nord- (Staatl. Materialprüfungsamt Nordrhein- len). *Arch. Eisenhüttenw.*, 1959, **30** (1), —The effects of the field of illumination, re, and inlet and exit slits on the determina- f line intensity and resolving power are sed. Conditions are given under which the ation of lenticular screens would be ad- geous. H. SAWISTOWSKI

. Use of direct-current arc in spectral analysis. d for complete evaporation of the sample. línek (Inst. of Metall., Acad. Sci., Prague). *Listy*, 1959, **53** (2), 145–153.—The principle method is given, with a survey of published unications dealing with its use. (29 refer- J. ZÝKA

4627. **Improved method of introducing [powdered] samples [into the arc] in spectrographic analysis.** O. F. Chesnokov and Yu. A. Kopeĭkin (Comprehensive Geophysical Expedition Sibreofiztrest). *Zavod. Lab.*, 1958, **24** (12), 1487–1489.—An electromagnetic vibrator is described. G. S. SMITH

4628. **Isotopic analysis by interferential spectrometry.** J. Artaud and S. Gerstenkorn (Commissariat à l'Énergie Atomique, Paris). Second U.N. International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Rep. A/CONF.15/P/328, 20 pp.—The principle of isotopic analysis by emission spectroscopy is discussed. The method is valid only for those elements where the isotopic displacement has a value twice as large as the Doppler broadening. The Fabry - Perot photoelectric spectrometer used for isotopic analysis is briefly described. The application of this method to the isotopic analysis of He, Li, Pb and U is described. NUCL. SCI. ABSTR.

4629. **Universal buffer solution for use in ultraviolet spectrophotometry.** M. T. Davies (British Drug Houses Ltd., Graham St., London). *Analyst*, 1959, **84**, 248–251.—Owing to the high light-absorbing properties of phenylacetic and diethylbarbituric acids, the buffer soln. of Prideaux and Ward (*J. Chem. Soc.*, 1924, **125**, 426) and Britton and Robinson (*Ibid.*, 1931, 458) require modification before they can be used in u.v. spectrophotometry. Aq. soln. of tri(hydroxymethyl)methylamine possess high uniform transmittance, and this base was found to be a suitable substitute for the barbitone of Britton and Robinson's mixture. The composition of the modified buffer soln. is given, and the pH values of liquids prepared by addition of 0·4 *N* HCl or 0·4 *N* NaOH to 50 ml of this soln. and dilution to 200 ml are tabulated. The values range from pH 2 to pH 12 and have been determined at 25° and at 37°. Ionic strength, dilution and buffer capacity data for these soln. are provided. A. O. JONES

4630. **Potassium bromide discs for mounting Nujol mulls.** Han Tai and A. L. Underwood (Dept. of Chem., Emory Univ., Atlanta, Ga., U.S.A.). *Chemist Analyst*, 1958, **47** (4), 104.—Nujol (liquid paraffin) mulls for i.r. spectroscopy are prepared by placing the sample (2 mg, 200 mesh) on the centre of a potassium bromide disc (200 mg, 0·5 in.), adding a drop of liquid paraffin ($\simeq$0·1 ml), covering with another disc, and rubbing the discs together to obtain a uniform mull. The disc sandwich is then mounted in a holder for examination. Alternatively, the sample may be mulled with a pestle and mortar, a drop of the mull placed between two discs and the discs moved about to give a uniform sample layer. The discs are rejected after use, and liquid paraffin spilled on the disc holder can be removed with CCl_4. This method gives spectra comparable in quality with those given by other mull methods. R. E. ESSERY

4631. **High-resolution infra-red grating spectrometer.** National Bureau of Standards (Washington, D.C., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (6), 175–176.—The spectrometer described provides extremely high resolution in the 3 μ to 6-μ region and can be used either as a single-pass or double-pass instrument. A special drive mechanism allows it to scan small portions of a rotational - vibrational band either quickly or slowly so that not only are the rotational lines resolved but the fine-structure

splitting of individual lines can be detected. By recording the absorption spectra simultaneously with the calibrated fringe system of a Fabry-Perot interferometer, wavelengths can be measured to 1 part in 5×10^5. K. A. PROCTOR

4632. **Use of crystals of silver chloride in infra-red spectrography.** Mlle Le Sech. *Chim. Anal.*, 1958, **40** (11), 425–429.—The advantages and disadvantages of the use of crystals of AgCl as transparent plates in the i.r. region beyond 9 μ are discussed. The material is sufficiently transparent from 0·6 to 25 μ and is resistant to acids, H_2O, CS_2 and hydrocarbon solvents. Its disadvantages are its high index of refraction, high ductility, ease of reduction by metals with which it comes in contact, sensitivity to visible and u.v. light, solubility in aq. NH_3 and other complexing agents, and high price. A special technique of pressing between perfect optical surfaces of plate glass or stainless steel is described, as normal polishing methods scratch it very easily. Examples of its use are given. The possibility of using thallium iodobromide is discussed.
E. J. H. BIRCH

4633. **Improved measuring apparatus for X-ray absorption spectrometry.** H. P. Hanson, W. E. Flynt and J. E. Dowdey (Dept. of Physics, Univ. of Texas, Austin, U.S.A.). *Rev. Sci. Instrum.*, 1958, **29** (12), 1107–1111.—Two methods of data-taking which eliminate the need for a stabilised X-ray power supply are described. In the first, the sample is oscillated in and out of the beam with a period small compared to drift. From the observed I and I_0 a quantity proportional to the absorption coefficient is electronically calculated and plotted. In the second, two counters are arranged in tandem with the sample between them, yielding I and I_0 simultaneously. G. SKIRROW

4634. **X-ray spectrographs for rapid X-ray fluorescence analysis with photographic recording.** E. F. Priestley (Min. of Supply, A.R.D.E., Fort Halstead, Kent, England). *J. Sci. Instrum.*, 1958, **35** (11), 409–413.—Curved-crystal spectrographs are described covering the range from K to U. The simultaneous recording of spectra between 0·5 and 2·5 A or 1·5 and 3·75 A is possible, and minor constituents (0·1%) can be detected. In more sensitive spectrographs (0·001%) a scanning device is used for covering the required wavelength range.
G. SKIRROW

4635. **Effect of variations in ambient temperature upon the optical alignment of an X-ray fluorescence spectrometer.** T. A. Davies (Mond Nickel Co., Ltd., Birmingham, England). *J. Sci. Instrum.*, 1958, **35** (11), 407–409.—The influence of temperature on the dispersion of an X-ray fluorescence spectrometer has been attributed to changes in the interplanar spacing of the diffracting crystal.
G. SKIRROW

4636. **Differential refractometer.** P. Kratochvíl and B. Sedláček (Phys. Chem. Dept., Chem. Inst. Acad. Sci., Prague). *Chem. Listy*, 1958, **52** (12), 2414–2416.—The suggested autocollimation arrangement enables a lens of large focal length to be used without lengthening the apparatus. The two-part tube permits the simultaneous control of the zero position as well as the location of the image. The accuracy can thus be increased. The light from the source passes through the filter and the rays are reflected from a small mirror to the axial direction of the apparatus. After passing through the tive (110 cm focal length), the rays pass thro tube, are reflected from the mirror and agai through the tube. The tube and mirror are in a double-jacketed protective vessel th statically controlled with H_2O. The arrang can be used for measurements of differences range of 1×10^{-2} units with an accuracy of $\pm$
J. Z

4637. **Automatic control of liquid compo based on variations of refractive index.** H. pecká and J. Koropecký (Inst. Chem. E Sch. of Chem. Technol., Prague). *Chem. Pr* 1958, **8** (12), 640–641.—The described meth based on the observation of the change of length of the lens-shaped tube through whic liquid flows. The sensitivity of this arrang has been tested with sugar soln. of different and it is claimed that the apparatus can be us measurements of n_λ^t with an accuracy $>$ 10^{-4} and that the relation between n_λ^t of the and the response of the recorder is linear wit range of 0·0800 unit of refractive index. apparatus can be adapted for the automatic c of production processes. J. Z

4638. **Microscope attachment for accurate m drilling and the removal of analytical samples small areas.** A. C. Simon and D. A. Gildner Naval Res. Lab., Washington, D.C.). *Rev. Instrum.*, 1958, **29** (12), 1125–1128.—The air-d micro-drill which can use drills as small as 0·0 in diam. is interchangeable with a micro objective and can be adjusted to work an centred under the cross-hairs of the obje The drilling process is observed from an aux stage with a low-power microscope, and the of the drill is controlled by the microscope adjustment. G. SKIRR

4639. **Ring-oven drop colorimetry with th of a standard silver sulphide scale.** H. V M. B. Čelap and V. V. Almažan (Inst. f. ano allg. Chem., Techn. Hochsch., Vienna). *Mikro Acta*, 1959, (1), 36–43.—Factors are given fo semi-quant. evaluation of the sulphide ring 14 ions obtained by the ring-oven techn The metals are fixed as sulphides, then conv into Ag_2S, and the intensity of the ring is com against a set of standards produced by heat series of CuS rings with 1% $AgNO_3$ soln.
T. R. ANDR

Electrical

4640. **Use of the pool cathode in polarogr** R. C. Rooney (Brit. Cast Iron Res. Ass., Bord Hall, Alvechurch, Birmingham, England). *Tal* 1959, **2** (2), 190–194.—A cell of the convent H type is illustrated, in which the difficult ensuring close reproducibility of the pool si overcome by forming the pool in a glass cup, v is filled to overflowing every time the pool is The advantages and disadvantages of this ce conventional and cathode-ray polarography discussed, and comparative results are quote three reactions in which the pool and drop mercury electrodes are used. In convent polarography, the pool electrode gives a gai sensitivity by a factor of 20 over the drop

ry electrode, but the fact that a repeat polaro- cannot be run renders it unsuitable for this f instrument. In cathode-ray polarography, ol electrode offers some advantages over the ng mercury electrode, the main disadvantage, ıch cell must be individually calibrated, or the ell used throughout, being less serious because pe of instrument produces polarograms every with a pool cathode, the peak height being ucible for 20 to 30 sweeps, which is ample or measuring the height. R. E. Essery

1. **Construction and characteristics of Teflon-d polarographic electrode for intra-vascular n determination.** J. Krog and K. Johansen Inst. for Exp. Med. Res., Ullevål Hosp., Norway). *Rev. Sci. Instrum.*, 1959, **30** (2), 09.—The Teflon membrane shows a high ability to O and can be heat-sterilised without ge. It shows good mechanical strength and stant to surface adhesions of proteins. It be free from pinholes, so that only cast Teflon s satisfactory. G. Skirrow

2. **Use of the rotating platinum micro-disc ode in polarographic analysis.** M. B. Bardin, . Lyalikov and V. S. Temyanko (Kishinevsk Univ.). *Zhur. Anal. Khim.*, 1959, **14** (1), —Distinct polarographic waves of good repro- lity were obtained for O, H^+, $[AuCl_4]^-$ and $]^{2-}$ with the rotating platinum micro-disc ode, in 0·1 *N* $NaNO_3$ as basal soln. In 10 gs (without cleaning the electrode) the max. ion from the average was $\gtrless$ 3%. For acy and reproducibility the electrode must be ately centred. The Levich equation was used calculations—

$$i_d = 0{\cdot}62\, nFD^{2/3}\omega^{1/2}\upsilon^{-1/6}cS,$$

i_d is the diffusion current, n is the number ectrons transferred in the reaction, F is ay's number, D is the diffusion coeff. in sq. r sec.; ω is the angular velocity of rotation m, where m is the number of revs. per sec.), inematic viscosity of the liquid in sq. cm per is the concn. of reducing ion in moles per and S is the electrode surface area in sq. cm. alues of i_d found for H^+ in a soln. 4×10^{-4} *N* Cl in a basal soln. of 0·1 *N* $NaNO_3$ differed the calculated values in 5 experiments at nt ω by 10·5 to 18·0%, which was con- d satisfactory agreement under the con- s used; S was 0·78 sq. mm. By using the n equation, the diffusion coeff. of $[AuCl_4]^-$ *N* $NaNO_3$ was calculated as $1{\cdot}2 \times 10^{-5}$ sq. r sec. C. D. Kopkin

3. **Transistorised switch for derivative polaro-y.** J. Krugers (Lab. for Anal. Chem., Univ. nsterdam, Holland). *Anal. Chem.*, 1959, **31** 44–447.—An electronic device is described to e mechanical switching. The circuit may e used for converting a small d.c. into an a.c. y desired frequency between 0·1 cycle and ocycles per sec. H. F. W. Kirkpatrick

4. **Automatic titrimetry: a simple apparatus ecording full-scale potentiometric titrations.** aslam and D. C. M. Squirrell (I.C.I. Ltd., cs Div., Welwyn Garden City, Herts., nd). *J. Appl. Chem.*, 1959, **9** (2), 65–68.— apparatus described consists of three main —(*i*) an injection delivery system (with hypo- c syringes of vol. 1, 5, 10 and 20 ml) that delivers titrant at a definite rate, (*ii*) a system indicating the change of pH or e.m.f. produced by the titrant, and (*iii*) a recorder with variable resistance to cover a range of 0 to 10 pH units or 0 to 50 mV. The chart speed of the recorder is 1 in. per min. High accuracy was attained in the following titrations—sebacic acid in nylon-610 salt cross-decomposed with *N* HCl; H_3PO_4 with NaOH soln.; chlorides; mixtures of chloride and bromide; amines with $HClO_4$ in glacial acetic acid; boric acid - mineral acid mixtures with NaOH; poly(methacrylic acid) in methyl methacrylate - methacrylic acid copolymers; salicylic acid in phenyl salicylate; and V^{3+} titrated with 0·02 *N* $2(NH_4)_2SO_4.Ce(SO_4)_2.2H_2O$. The calibration of the hypodermic syringes should be checked frequently, whilst the titrant soln. should be standardised against a known amount of pure compound.

W. J. Baker

4645. **Potentiometric micro-titration apparatus.** I. D. P. Wootton (Post-grad. Med. Sch., Hammersmith Hosp., London). *Lab. Practice*, 1959, **8** (1), 11–13.—The titration assembly and electronic potentiometer described and illustrated are used for the routine determination (by $AgNO_3$ titration) of Cl^- in samples (0·1 or 0·2 ml) of plasma, serum, urine, or other fluids, by the method of Sanderson (*Biochem. J.*, 1952, **52**, 502). Reproducibility is within 1 milli-equiv. per litre for plasma levels of $\simeq$ 100 milli-equiv. per litre. The values obtained are claimed to be the true content of Cl^-, those obtained by the Van Slyke method being low by 2 to 3 milli-equiv. per litre. W. J. Baker

4646. **Anodic chronopotentiometry at solid electrodes. Precision as a function of transition time for some organic compounds.** J. D. Voorhies and N. H. Furman (Frick Chem. Lab., Princeton Univ., N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 381–384.—A simple oscillographic method involving Polaroid photographic recording has been used to measure short transition times in chronopotentiometric analyses and has shown that certain time-dependent errors can be eliminated by using such short transition times. K. A. Proctor

4647. **Use of dibromohydroxyquinoline in amperometric titration.** L. I. Panteleeva. *Nauch. Zap. L'vovsk. Politekh. Inst.*, 1958, (50), 50–56; *Ref. Zhur., Khim.*, 1959, (5), Abstr. No. 15,075.—The titration of Fe, Cu and Ti with a dropping mercury cathode is based on the pptn. of these metals with an acid soln. of 35% 5:7-dibromo-8-hydroxyquinoline (**I**) in acetone. Titration of Cu and Fe is carried out without the application of an external e.m.f.; the reducing current is applied to a metal soln. at 50°, containing 10% (v/v) of acetone and 1% gelatin soln. (2 to 3 drops); there is no preliminary removal of O. Mercury is extracted from the soln. with a small amount of $CHCl_3$. Titration of $>$ 0·04 mg of Fe per 10 ml in 0·01 to 0·02 *N* HCl or H_2SO_4 is not affected by alkali metals, alkaline-earth metals, Be, Mn, Al, Cr, As, Sb, Sn, Cd, Zn or Hg, as these are not pptd. by **I** in acid medium; Ti is complexed with tartaric or citric acid; Cu and V do not interfere. Copper is titrated in 0·01 to 0·05 *N* HCl or H_2SO_4; a 300-fold excess of Al does not affect the titration; $>$ 0·5 mg of Cu in the presence of $<$ 20 mg of Fe can be titrated in the presence of ammonium oxalate (5 mg for each mg of Fe) at pH 2 to 4 with an accuracy of $\simeq$2%. *Procedure for Cu in aluminium alloys*—Dissolve the sample (0·5 to 1 g) in HCl (30 ml) or in HNO_3 (1:1) (1 ml)

evaporate almost to dryness, dissolve in H_2O and make up to 100 ml with H_2O; to a 2 to 5-ml aliquot in the titration vessel add a little $CHCl_3$, acetone (1 to 2 ml) and 1% gelatin soln. (2 to 3 drops) and make up to 15 ml with ammonium oxalate soln. (2 g in 100 ml of 0·1 *N* HCl); place in a thermostat at 50° for 10 min. and titrate with **I**. The determination takes 90 min. *Procedure for Ti*—Determine Ti by back-titration of an excess of **I** with a copper soln. Make the test soln. 0·01 to 0·015 *N* with respect to HCl or H_2SO_4 and 10% with respect to acetone, heat to 50° to 60°, add a soln. of **I** dropwise with stirring and, when the TiO-$(C_9H_4Br_2ON)_2$ is completely pptd., add an excess of **I** (2 to 3 ml) and back-titrate amperometrically with standard copper soln. If the concn. of acetone in the soln. is $> 30\%$ add hot 0·1 *N* HCl. K. R. C.

4648. **Activation analysis for industries.** A. De Haan, jun. (Tracerlab, Inc., Richmond, Calif.). *A.S.T.M. Special Tech. Publ.*, 1958, (215), 54–61.—The basic principles and techniques of activation analysis are discussed. Applications are cited to the analysis of trace concn. of chemical elements in metals and alloys, in chemicals, petroleum, food, plastics and other industrial products.

CHEM. ABSTR.

4649. **Radiochemical tracer analysis. A new approach resulting in increased accuracy.** D. A. Lambie (U.K.A.E.A., Radiochem. Centre, Amersham, Bucks., England). *Analyst*, 1959, **84**, 173–176.—It is shown that the errors inherent in radioactive tracer analysis may be reduced by measuring the radioactivity of the unseparated fraction (*i.e.*, the filtrate) rather than that of the separated fraction (*i.e.*, the ppt.). A satisfactory application to the determination of Co in the presence of Ni is described. A. O. JONES

4650. **Particle-size analysis by gamma-ray absorption.** C. P. Ross (Savannah River Lab., E. I. du Pont de Nemours & Co., Inc., Aiken, S.C., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 337–339.—The concn., at a fixed depth, of uranium oxide particles settling from a uniform suspension was continuously determined by measuring the transmittance of γ-rays from a ^{241}Am source. For the particles tested, the absorption of γ-rays depended only on the mass of suspended material, and the data obtained were converted to a standard curve of particle fractions below a certain size *vs.* the diameter obtained from Stokes's law. The fraction undersize was determined with a 95% confidence limit of $\simeq \pm 0{\cdot}04$ and the average diameter according to Stokes's law was determined to about $\pm 0{\cdot}3$ μ. The method is applicable to particles up to 74 μ in diameter that have an absorption coeff. significantly diff from that of the suspending medium.

K. A. PROCT

4651. **Rapid analysis of unknown mixed γ-em with a γ-ray scintillation spectrometer.** F Aoki, Seishi Yajima and Toshio Kurosawa (J Atomic Energy Res. Inst., Tokai, Ibaragi-*Japan Analyst*, 1958, **7** (11), 695–699.—Fi products were fractionated by the ion-exch resin method (Cohen *et al.*, *Nucleonics*, 194 22; Thompkins *et al.*, *J. Amer. Chem. Soc.*, **69**, 2969) and each fraction was examined w γ-ray scintillation spectrometer for the ap amount of each fission product to be determ

K. SA

4652. **Note on a possible source of error in cou radioactive samples.** G. Saucier and L. P. I (Dept. Biol., Univ., Ottawa, Ont.). *Canad Biochem. Physiol.*, 1959, **37** (5), 721–722.—In course of an experiment in which ^{14}C was inco ated into the cholesterol in different tissues, a of activity occurred when samples of digit were re-counted some hours later. This eff ascribed to the natural radioactivity presen atmospheric dust. N.

4653. **Method and apparatus for analy mixtures of gaseous materials by compos modulation.** Esso Research and Engineering Brit. Pat. 814,317; date appl. 25.4.56. U. date appl. 20.5.55.—The key component in a st of a (binary) mixture of gaseous materials is d mined by passing the stream at const. temp. an adsorber in which the pressure is v cyclically, between predetermined upper and l limits, for a time period, at each pressure, suffi for the adsorbent to reach an equilibrium cap for the key component, so that its conte cyclically and alternately modulated to dec the content at the higher pressure and increa at the lower pressure. The range of modulati the key component in the stream is detected electrically indicated with reference to that identical stream containing a known amount o key component. J. M. JACO

4654. **Combustible-gas analysers for high-perature operation.** N. W. Hartz. *Instrum. Co Systems*, 1959, **32** (6), 886–887.—An instrume described for continuous monitoring of inflamm vapours in ovens drying coated fabrics, abr products, brake linings, cast plastic films, etc. N.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any ubt might arise from the use of an abbreviation or symbol the word is printed in full.

ternating current	a.c.
mpere	amp.
ngström unit	Å
nhydrous	anhyd.
pproximate, -ly	approx.
queous	aq.
tmospher-e, -ic	atm.
oiling-point	b.p.
ritish thermal unit	B.Th.U.
alorie (large)	kg-cal.
alorie (small)	g-cal.
entimetre	cm
oefficient	coeff.
oncentrated	conc.
oncentration	concn.
onstant	const.
orrected	(corr.)
rystalline	cryst.
rystallised	cryst.
ubic	cu.
urrent density	c.d.
ycles per second	c/s
ensity	ρ
ensity, relative	d or wt. per ml
ilute	dil.
irect current	d.c.
istilled	dist.
thylenediaminetetra-acetic acid	EDTA
lectromotive force	e.m.f.
quivalent	equiv.
ram	g
ram-molecule	mole
alf-wave potential	$E_{\frac{1}{2}}$
our	hr.
ydrogen ion exponent	pH
nfra-red	i.r.
nsoluble	insol.
nternational unit	i.u.
ilogram	kg
ilovolt	kV
ilowatt	kW
iquid	liq.
naxim-um, -a	max.
nelting-point	m.p.
nicrogram	μg (not γ)
nicrolitre	μl
nicromole	μmole
nicron	μ
nilliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_l^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm.
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or n mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
s proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the dentifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances n the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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